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AMMRC CTR 75-6

DEVELOPMENT AND EVALUATION OF PHOSPHONITRILIC FLUOROELASTOMER O-RINGS

APRIL, 1975

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THE FIRESTONE TIRE & RUBBER COMPANY
AKRON, OHIO 44317

FINAL REPORT, CONTRACT DAAG46-74-C-0066

Approved for public release; distribution unlimited.

Prepared for

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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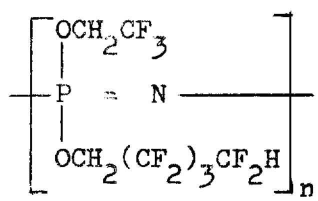
ABSTRACT

The purpose of this investigation was to develop and evaluate phosphonitrilic fluorelastomer compounds for O-ring hydraulic seal applications. Formulations were sought which would be serviceable in hydraulic fluids over the temperature range of -80°F to 400°F (-62°C to 204°C).

The polymer used in this investigation was a phosphonitrilic fluoroelastomer prepared in the Central Research Laboratories of The Firestone Tire & Rubber Company and having the following formula:



The polymer contained sufficient cure sites to attain good curability with conventional peroxide curatives.

In addition to the low temperature flexibility indicated above the following target values were adopted for this investigation: Tensile strength - 1500 psi, elongation at break - 125%, 100% modulus - 800 psi, Shore hardness - 70, compression set (70 hrs.@ 300°F) - 20%.

This investigation was conducted under Contract No. DAAG46-74-C-0066 from the U. S. Army Materials and Mechanics Research Center, Watertown, Massachusetts 02172. The effects of reinforcing agents, vulcanization agents and stabilizers on compound properties were investigated. Efforts were made to optimize curing times and temperatures; stress-strain properties; hardness; compression set; tear and abrasion resistance; fluid, water, steam and acid resistance, and low temperature flexibility.

In addition, the Seal Group of Parker Hannifin fabricated O-ring seals from selected stocks and conducted dynamic extrusion and chew tests on the seals. These tests showed the best phosphonitrilic fluoroelastomer O-ring compound to be the following:

Polymer K-17638	100.0 parts
Quso WR 82	30.0 parts
Stan Mag ELC	6.0 parts
Stabilizer - $(8HQ)_2Zn$	2.0 parts
Union Carbide Silane A-151	2.0 parts
Vulcup R	0.4 parts

This formulation afforded the best balance of stress-strain properties, hardness, compression set resistance, hydraulic fluid resistance and heat resistance. O-rings fabricated from this formulation should be serviceable for extended times over the temperature range of -70° F to 350° F (-57° C to 177° C).

In related studies experiments were conducted on the coating of stainless steel cable with phosphonitrilic fluoroelastomer compounds. A good quality coating of approximately 0.031" thickness was obtained by passing the cable through a crosshead extruder followed by vulcanization of the coating for 1 minute at 392° F (steam).

Phosphonitrilic fluoroelastomer compounds show limiting oxygen index (LOI) values of 50-60 depending on the type and level of filler incorporated in the compounds. These high LOI values add still another dimension to the applicability of phosphonitrilic fluoroelastomers in highly sophisticated environments.

This investigation has clearly established that phosphonitrilic fluoroelastomers have potential for applications demanding extreme low temperature flexibility, outstanding fluid resistance, good heat resistance and good dynamic properties. O-ring seals are one such application for which no existing commercial elastomer currently has met the full range of properties required.

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INTRODUCTION

Phosphonitrilic fluoroelastomers are a new class of petroleum resistant polymers which exhibit excellent low temperature flexibility and solvent resistance and good heat resistance. Preliminary compounding studies have demonstrated that phosphonitrilic fluoroelastomers can be vulcanized and reinforced with conventional agents to give high quality materials [1].

In earlier work, phosphonitrilic fluoroelastomers were used for fabrication of Arctic fuel hose [2], lip seals [3], and for O-ring seals and gaskets [4,5]. This investigation was directed toward the further development of phosphonitrilic fluoroelastomers for O-ring hydraulic seal applications requiring servicibility over the temperature range of -80°F to 400°F (-62°C to 204°C).

This work was conducted under Contract No. DAAG46-74-C-0066 from the U. S. Army Materials and Mechanics Research Center (AMMRC), Watertown, Ma. 02172. The rubber utilized in this study was phosphonitrilic fluoroelastomer supplied to AMMRC by The Firestone Tire and Rubber Company under Contract No. DAAG46-74-C-0047.

The phosphonitrilic fluoroelastomer utilized in this investigation had the following general formula:

$$-\left[\begin{array}{l} OCH_2CF_3 \\ \quad | \\ P = N - \\ \quad | \\ OCH_2(CF_2)_3CF_2H \end{array} \right]_n-$$

Sufficient cure sites to achieve good curability with conventional peroxide curing agents were incorporated into the polymer. The polymers in Table I differed only in cure site level.

This type of polymer is prepared by thermal polymerization of cyclic chlorophosphazene trimer and tetramer to yield a soluble linear polydichlorophosphazene. The reactive chlorine-phosphorus bonds in the chloropolymer are then substituted with the appropriate fluoroalkoxide mixtures to yield the desired phosphonitrilic fluoroelastomers.

These elastomers are characterized by very low glass transition temperatures, excellent resistance to hydrocarbon fluids, resistance to hydrolysis and good resistance to thermal degradation or crosslinking.

Phosphonitrilic fluoroelastomers respond well to reinforcement with reinforcing agents such as silicas or carbon blacks. Conventional peroxide curing agents can be utilized to develop a range of vulcanizate properties which make these elastomers suitable for a broad spectrum of applications.

This report summarizes work which was performed to further investigate the properties of phosphonitrilic fluoroelastomer vulcanizates and to optimize the properties for use in O-ring hydraulic seals. The target values for physical properties critical to O-ring performance were: tensile strength - 1500 psi, 100% modulus - 800 psi, elongation at break - 125%, compression set (70 hrs.@300°F) - 20% and hardness - 70 Shore A. A dynamic testing program was utilized to further evaluate the performance of vulcanizates which appeared to have potential in O-ring hydraulic seals.

SUMMARY

Phosphonitrilic fluoroelastomers were compounded in a variety of formulations in an effort to produce O-ring seals which would perform for extended periods in hydraulic fluids over the temperature range of -80°F to 400°F. Several types and levels of fillers were evaluated in conjunction with a variety of peroxide curing agents in phosphonitrilic fluoroelastomers to provide stocks having optimum stress-strain properties, hardness, compression set resistance, tear and abrasion resistance, heat resistance, hydraulic fluid resistance, chemical resistance and maximum low temperature flexibility.

A significant effort to optimize processibility of the stocks in conventional rubber processing equipment was also made. Various mixing conditions and processing additives were investigated.

Stocks were developed to yield economically feasible cure times in existing commercial O-ring fabrication processes. The judicious choice of curing agents, curing temperatures, and curing times yields stocks which process quite acceptably without major modifications of existing O-ring fabrication processes.

The compounding studies conducted under this contract have shown that silane-treated silica reinforcing agents provide compounds with better heat resistance than compounds filled with carbon blacks. Phosphonitrilic fluoroelastomer vulcanizates appear to have better thermal stability in the presence of inorganic fillers than with carbon blacks. No mechanistic interpretation for these facts is available at the present time.

The very demanding mechanical property requirements for O-ring hydraulic seal applications suggested the use of highly reinforcing fillers such as silicas and carbon blacks. The current investigation concentrated on evaluation of vulcanizates containing these two classes of fillers rather than the less reinforcing silane treated clays, although the latter fillers could conceivably be utilized as well.

On the basis of overall mechanical properties and heat resistance, Quso WR 82 reinforcing silica appears to be the best filler evaluated to date for phosphonitrilic fluoroelastomer O-rings. For optimum cures in O-ring compounds, Vulcup R gives the best balance of properties.

The optimum O-ring formulation developed under Contract No. DAAG46-74-C-0066 is as follows:

Polymer K-17638	100.0 parts
Quso WR 82	30.0 parts
Stan Mag ELC	6.0 parts
Stabilizer $(8HQ)_2Zn$	2.0 parts
Union Carbide Silane A-151	2.0 parts
Vulcup R	0.4 parts

Phosphonitrilic fluoroelastomer O-rings fabricated from this formulation exhibit good mechanical properties (abrasion and extrusion resistance) and heat resistance (to 350° F), excellent hydraulic fluid resistance, and excellent low temperature flexibility to (-70° F).

This study has clearly demonstrated the applicability of phosphonitrilic fluoroelastomers in high performance seals. Further development of these materials should provide O-rings with outstanding service life in very demanding applications.

RECOMMENDATIONS

Further development is required to fully realize the outstanding potential of phosphonitrilic fluoroelastomers in high performance hydraulic fluid O-ring seals. The following aspects of phosphonitrilic fluoroelastomer compounding should be evaluated further:

1. Improved interaction between polymer and filler should provide higher modulus, better compression set resistance, higher abrasion resistance and a higher temperature limit. A variety of silane coupling agents and reinforcing silicas should be evaluated to find the optimum combination for phosphonitrilic fluoroelastomers.
2. Additional additives for improved mixing and processing in conventional rubber machinery should be evaluated.
3. The mechanism of the degradative process which results in the present 350°F service limit for phosphonitrilic fluoroelastomer O-rings should be elucidated. Once this mechanism is elucidated, additives to retard the degradative process should be investigated.
4. An extensive program of fabrication of phosphonitrilic fluoroelastomer O-rings by injection molding and compression molding should be conducted. These O-rings should be evaluated in field tests to fully determine their strengths and weaknesses under actual service conditions.

INVESTIGATION

I. SUMMARY OF FIRST QUARTER RESULTS AND PROGRAM FOR SECOND QUARTER

The first quarter effort was directed toward "screening" of five carbon black and five silica reinforced vulcanizates. All of these stocks were cured with dicumyl peroxide and contained polymer K-17217 which is described in Table I. The results of these studies are summarized in Tables II and III. A brief summary of the conclusions from these studies are as follows:

A. Carbon Black Reinforced Stocks (R-190,264--190,268)(Table II)

1. Mill Processing -- All stocks exhibited stick to mill rolls at 130°F. The addition of low MW polyethylene (3 phr) Epolene C-305-G (R-190,265) provides a slight improvement in mill release.

2. Cure Profile -- All stocks showed good scorch safety at 335 and 370°F. The time to optimum cure at 370°F ranges from 3.0-4.0 min. and is suitable for factory O-ring cure cycles.

3. Stress-Strain Properties (30' @ 320°F) are suitable for all stocks except R-190,266 which exhibited fairly low tensile strength (FEF-MT black reinforcement). Higher values for the 100% Modulus would be desirable for all stocks. Press cures at 370°F utilizing optimum cure times result in lower cure states. It may be necessary to add additional peroxide for these higher temperature cures.

4. Aged Stress-Strain -- These stocks exhibit good retention of stress-strain properties after aging 240 hrs. at 275 or 300°F in air. However, after 240 hrs. at 350°F a significant loss in modulus and tensile strength was observed. After aging in hydraulic fluid (Mil-H-5606) for 240 hrs. at 73°F, these stocks showed essentially no change in stress-strain properties. However, after aging 240 hrs. at 275°F, the stocks showed considerable softening. The use of stabilizer (1 phr of $(8\text{-HQ})_2Zn$ in R-190,264) results in a modest improvement in thermal stability compared to the control (R-190,268).

5. Normal and Aged Shore A Hardness -- The hardness values ranged from 43-47 durometer. These values are much too low for O-ring stocks (70 durometer required), therefore some means for increasing hardness must be developed. Air aging for 240 hrs. at 275°F generally results in a slight increase in hardness. Aging 240 hrs. at 73°F in hydraulic fluid results in essentially no change in hardness, while at 275°F a substantial softening is observed.

6. Compression Set (70 hrs. @ 275°F) -- Compression set values ranged from 21 to 31% for cylinders and from 26-48% for plied disks. Stock R-190,267 (FEF-Austin black) gave the lowest value, 21%.

7. Tear Strength -- The values ranged from 27 to 92 ppi. Stock R-190,267 (FEF-Austin black) gave the highest value (92 ppi) while R-190,266 (FEF-MT black) gave the lowest (27 ppi). All of these stocks exhibit fairly low tear strength, and considerable work is needed to improve this property.

8. Low Temperature Properties -- The Gehman T_5 values ranged from -51 to -62°F. The YMI* values were essentially the same (-59 to -63°F). Figures 1 and 2 show Gehman Twist and Gehman Flexure for stock R-190,266. Table XIX shows the Gehman data for this stock.

9. Resistance to Hydraulic Fluid (Mil H-5606) -- All of these stocks exhibit excellent solvent resistance after aging 240 hrs. at both 73 and 275°F. Actually, these stocks do not exhibit enough volume swell since ca. +5% is preferred.

B. Silica Reinforced Stocks (R-190,279 - 190,283)(Table III)

1. Mill Processing -- All of these stocks exhibit better mill processing, i.e., less stick to mill and better green strength than the carbon black reinforced stocks. The addition of Epolene-305-G (1 phr) (R-190,280) provides a slight improvement in mill release.

2. Cure Profile -- These stocks exhibit good scorch safety at 335 and 370°F. However, the optimum cure times at 370°F are relatively long (6.6 to 18.3 min.) for factory O-ring cure cycles. It will be necessary to find some means of activating the cure at 370°F to produce shorter cure times.

* YMI represents Young's Modulus Index (the temperature (°C) at which the Young's Bending Modulus (ASTM D-797) reaches 10,000 psi).

3. Stress-Strain Properties -- Press cures of 30 min. at 300°F resulted in good stress-strain properties for all stocks. Slightly higher values for the 100% modulus would be desirable. Press cures at 370°F using optimum cure times resulted in much lower cure states. It may be necessary to adjust the peroxide level for the 370°F cure.

4. Aged Stress-Strain -- These stocks exhibited good retention of stress-strain properties after aging 240 hrs. at 275 and 300°F in air and fair retention after 240 hrs. at 350°F in air. Good retention of properties was observed after 240 hrs. at 73°F or 275°F in hydraulic fluid. In general these silica reinforced stocks showed much better heat-aging properties than do carbon black reinforced stocks.

5. Normal and Aged Shore A Hardness -- Hardness values, ranging from 58 to 65 durometer, are considered adequate for most O-ring applications. These stocks exhibit good retention of hardness on aging 240 hrs. at 275°F in air and 240 hrs. at 73°F in hydraulic fluid.

6. Compression Set (70 hrs. @ 275°F) -- % Set values ranged from 54 to 69% for cylinders and from 62 to 76% for plied disks. These values are much higher than observed for carbon black reinforced stock and also too high for most O-ring applications. Further studies should be directed toward screening of silicas of larger particle size and lower surface area.

7. <u>Low Temperature Properties</u> -- Gehman T_5 values ranged from -45 to -51°F while YMI ranged from -55 to -57°F. These values are 6 to 11°F higher than corresponding values for carbon black reinforced stocks.

8. <u>Resistance to Hydraulic Fluid (Mil-H-5606)</u> -- These stocks exhibit excellent resistance to hydraulic fluid after aging 240 hrs. at 73°F and 275°F. The % volume swell value observed for R-190,280 (240 hrs. @ 275°F) (26.15%) is in question in view of the low value obtained for 240 hrs. at 73°F (1.49%). As with the carbon black reinforced stocks, these compounds are too resistant to hydraulic fluid. Some means should be developed for increasing volume swell to <u>ca.</u> +5%.

C. <u>Silica and Carbon Black Reinforced O-Ring Compounds -- Attempts To Improve Hardness of Carbon Black Reinforced Stocks and Evaluation of Quso WR-82 Reinforced Stocks (Table IV)</u>

Compound R-191,920 was a standard FEF black, peroxide cure type formulation containing a crosslink promoter, Chem Link 30 (3 phr). This stock appears to be highly overcured, but the optimum cure at 370°F may afford better properties. This stock exhibits adequate scorch safety at 335 and 370°F and an acceptable cure time at 370°F (5.0 min.).

The addition of a processing aid, AC polyethylene (5 phr) (R-191,921), improves the mill release of the stock. This processing aid also lowers the cure state so that this stock has excellent stress-strain properties. Compound R-191,921 shows good scorch resistance at 335 and 370°F and an acceptable cure time at 370°F (4.3 min.).

Compound R-191,922 contained FEF black (25 phr) and graphite (15 phr) with a peroxide cure. This stock appears to be highly overcured but still shows good tensile strength. This compound has excellent scorch resistance at 335 and 370°F and an acceptable cure time at 370°F (4.5 min.).

Compound R-191,924 contains Quso WR-82 (25 phr) with a peroxide cure. This stock appears to be highly overcured as evidenced by stress-strain data. This compound shows good scorch resistance at 335 and 370°F and a relatively long cure time at 370°F (14.6 min.). Final conclusions on these stocks will be made when all physical tests have been completed.

D. O-Ring Stocks for Parker Seal

After evaluation of the physical testing data on the five carbon black reinforced O-ring formulations, the FEF-Austin black compound was selected to be submitted to Parker Seal (R-191,941)(Table V). However, all of the five silica-reinforced compounds gave compression set values that were too high for O-ring applications. Recent studies, outside the O-ring contract, revealed that Quso WR-82 (organosilylated silica of larger particle size and lower surface area than Silanox 101) gave stocks with much lower compression set

Parker Seal suggested that the following physical property improvements in the O-ring compounds should be given first priority:

1. Improve processing via silicone rubber addition.
2. Increase hardness.
3. Improve compression set.
4. Develop compounds that have high low strain (25%) modulus -- low strain modulus should increase slowly with aging.
5. Evaluate silane coupling agents.

A large batch (1 lb.) of Quso WR-82 reinforced stock will be sent to Parker Seal for the Second Quarter. Also, a large batch (1 lb.) of the same compound containing 15 phr Silastic 410 is being submitted to determine the effect of silicone rubber on O-ring seal performance. Parker Seal will then construct O-rings (size 214) and run the following tests:

1. Effect of cure and post cure conditions on O-ring performance.
2. Dynamic "Chew Tests" on phosphonitrilic fluoroelastomer and fluorosilicone compounds under more severe conditions -- longer times at 302°F and 350°F.
3. Extrusion tests on phosphonitrilic fluoroelastomer and fluorosilicone seals.

Appendix II contains the Status Report of the First Quarter material as provided by Parker Seal Company.

B. Second Quarter Evaluation of Phosphonitrilic Fluoroelastomer O-Ring Compounds

1. Quso WR-82 Reinforced O-Ring Compounds (Table VI)

Quso WR-82 reinforced phosphonitrilic fluoroelastomer O-ring compounds were found to have excellent heat-stability and compression set properties in previous studies (Tables IV and V). In compounds R-191,972 and 975 the Quso WR-82 level was increased from 25 to 40 phr while maintaining a constant level of the other compounding ingredients. Compound -976 was the same as -973 except for the inclusion of an additional 1.0 phr of stabilizer. The effect of varying Quso WR-82 and stabilizer levels on cure, mechanical and heat-stability are now summarized.

a. Mill Processing (130°F) -- All compounds are sticky on a warm mill and tend to split to both rolls. The compounds become less sticky as the Quso WR-82 level is increased but are still difficult to process at the highest level.

b. Cure Profile -- All compounds have good scorch safety at 335°F. The cure times at 370°F, 8.6 to 30.3 min., are too long for factory O-ring press cure cycles (ca. 5 min.). Some means of accelerating the peroxide vulcanization of these stocks should be developed.

c. Stress-Strain Properties -- The 50% moduli appear to increase as the silica level is increased from 25 to 30 phr then level off at ca. 575 psi. The 100% modulus and tensile strength increase as the silica level is raised from 25 to 30 phr then decreases as the level increases to 40 phr. The elongation at break decreases and the % tension set increases with increasing levels of Quso WR-82. These changes in stress-strain properties are typical for increasing levels of a reinforcing filler. The best overall stress-strain properties appear to be obtained at a silica level of 30 phr (R-191,973). The addition of 1 phr of stabilizer to -973, i.e., R-191,976, appears to have little effect on stress-strain properties except for a slight lowering of modulus. The press cures @ 370°F under optimum cure times resulted in a substantial decrease in cure state of these stocks.

d. Aged Stress-Strain -- All stocks have good retention of stress-strain properties after aging 672 hrs. @ 275°F in air, 240 hrs. @ 300 and 350°F in air and 240 hrs. @ 275°F in hydraulic fluid. Aging of stocks out to 672 hrs. @ 300 and 350°F in air and @ 275°F in hydraulic fluid are still in progress at the present time, and these data will be reported at a later date. The addition of extra stabilizer (1 phr), compound -976, does not result in any improvement in retention of modulus, but a substantial improvement in tensile strength retention is realized. O-rings constructed from any of these compounds should still function after 240 hrs. @ 300°F (air).

e. Shore A Hardness -- As the Quso WR-82 level is increased from 25 to 40 phr, the hardness is raised from 47 to 72 durometer. The Quso WR-82 level must be in the range of 30-40 phr to obtain O-ring compounds with suitable hardness. These compounds maintain excellent retention of hardness after aging in air at 275-350°F and in MIL-H-5606-C at 275°F.

f. Compression Set (70 hrs. @ 275°F) -- The values ranged from 20 to 42% for cylinders and 34 to 66% for plied disk. The compression set increases, as expected, as the silica level is raised from 25 to 40 phr. The maximum amount of Quso WR-82 in the compounds should be limited to the 30-35 phr range to maintain compression set in the range required for O-ring applications.

g. Tear Strength -- The tear strength increases from 58 to 121 ppi as the Quso WR-82 level is raised from 25 to 40 phr as would be expected for the addition of a reinforcing filler.

h. Abrasion Resistance -- The Abrasive Indices decreased from 64 to 42 as the Quso WR-82 level was increased from 30 to 40 phr. This decrease in abrasion resistance appears to result from the decrease in tensile strength of these compounds as the silica level increases (overloaded). The

Abrasive Index of -976 (86) is substantially higher than that of -973 (64) which contains 1 phr less stabilizer. These stocks are considered to have fair-good abrasion resistance.

i. Low Temperature Properties -- The Gehman T_{10} values were all essentially -50°C (-58°F), thus indicating that seals fabricated from these compounds should function down to -65°F. The YMI values ranged from -57°C (-71°F) to -61°C (-78°F). The level of Quso WR-82 appears to have very little effect on low temperature flexibility.

j. Resistance to Hydraulic Fluid (MIL-H-5606-C) -- All of these compounds exhibited excellent resistance to this hydraulic fluid after 240 hrs. @ 275°F, i.e., volume swell of less than 3%.

In summary these Quso WR-82 reinforced stocks have excellent stress-strain, heat-stability, compression set, low temperature flexibility and hydraulic fluid resistance and fair-good tear and abrasion resistance. In consideration of overall mechanical properties, the best level of Quso WR-82 appears to be 30-35 phr.

2. Quso WR-82 Reinforced O-Ring Compounds -- Study of the Effects of Peroxide Level, Different Cure Activator and Small Amounts of Reinforcing Silicas (Table VII)

Compounds R-191,977 and -978 were identical except for the addition of 0.5 phr Dicup 40C in the former and 1.5 phr in the latter. These can be compared to R-191,973 (Table VI) which contained 1 phr of Dicup 40C. These compounds were formulated to determine the effect of peroxide level on physical properties. Compound -979 was identical to -973 (Table VI) except Stan Mag ELC was replaced by a new acid acceptor, Tribase, in an attempt to increase the cure rate @ 370°F. Compound -980 contained Silanox 101 (10 phr) which replaced the corresponding amount of Quso WR-82 in an attempt to improve tear and abrasion resistance. Compound -981 contained Cab-O-Sil S-17 (5 phr) which replaced the corresponding amount of Quso WR-82 in an attempt to increase tear and abrasion resistance. These last two compounds should be compared to -973 (Table II).

a. Mill Processing -- All compounds were difficult to process on a 130°F mill, i.e., sticky and split to both rolls.

b. Cure Profile -- All of these compounds have good scorch safety @ 335°F. However, the optimum cure times @ 370°F for compounds -977 and -978 are much too long, 14.3 and 21.3 min., respectively. The replacement of Stan Mag ELC with Tribase, compound -979, results in a significant increase in cure rate @ 370°F, i.e., cure rate index increases from 8.4 (-973) to 20.0 (-979). This compound (-979) then has an acceptable cure time @ 370°F, 6.5 min. The replacement of 10 phr of Quso WR-82 with Silanox 101 (-980) results in a tremendous increase in the cure rate index, 8.4 (-973) to 83.3 (-980) and an acceptable cure time of 3.5 min. The replacement of 5 phr of Quso WR-83 with Cab-O-Sil S-17 (-981) results in a decrease in the cure rate index, 8.4 to 3.5 and an unacceptable cure time of 29.8 min.

c. Stress-Strain -- As the peroxide level is increased from 0.5 phr (-977) to 1.0 phr (-973) to 1.5 phr (-978), there is a corresponding increase in modulus and a decrease in elongation and tension set. The tensile strength changes only slightly with increasing peroxide level. The best peroxide level for O-ring compounds would be in the 0.5 to 1.0 phr level since 1.5 phr peroxide results in overcure (-978). The replacement of Stan Mag ELC by Tribase (-979) results in a slightly lower cure state (-973) but additional peroxide could adjust the cure to the same level. The replacement of 10 phr Quso WR-82 with the corresponding level of Silanox 101 (-980) results in lower 50% modulus, higher 100% modulus, tensile strength and elongation and a decrease in tension set. The replacement of 5 phr Quso WR-82 with the corresponding amount of Cab-O-Sil S-17 (-981) results in an increase in 50 and 100% moduli, tensile strength and elongation and tension set. Press cures of tensile slabs under optimum conditions @ 370°F resulted in substantially lower cure states. It may be necessary to adjust peroxide level and/or cure times for stocks cured @ 370°F.

d. Aged Stress-Strain -- Compounds -977, -973, -978 exhibited excellent heat resistance after 240 hrs. @ 275, 300 and 350°F in air and 240 hrs. @ 275°F in MIL-H-5606-C. The effect of increasing peroxide level is to maintain a higher modulus after a given aging time. Evidently, the crosslinks are quite thermally stable. Compound -979 appears to be essentially equivalent to -973 in heat-stability except for 350°F. After 240 hrs. @ 350°F this stock had completely degraded via a softening effect which precludes the use of this compound for high temperature O-ring applications. Compounds -980 and -981 appear to have essentially the same heat-stability as -973 (Table VI). All of these compounds, except -978, should be suitable for O-ring applications in the 275-350°F range. Additional heat-aging studies are in progress and will be reported at a later date.

e. Shore A Hardness -- As the Dicup 40C is increased from 0.5 to 1.5 phr, the hardness is raised from 45 to 56 durometer. The use of Tribase in place of Stan Mag ELC (-979) results in a slightly lower hardness (49)(compare to -973, Table VI). The use of 10 phr of Silanox 101 in place of Quso WR-83 in -980 also results in a slight lowering of hardness (49). However, the use of only 5 phr of Cab-O-Sil S-17 in place of the corresponding amount of Quso WR-82 results in a large increase in hardness (66 durometer). All of these compounds exhibit excellent retention of hardness after aging 675 hrs. @ 275°F (air), 240 hrs. @ 300 and 350°F (air) and 240 hrs. @ 275°F in MIL-H-5606-C.

f. Compression Set (70 hrs. @ 275°F) -- Most of the compounds are still in the process of being tested and data will be reported at a later date. The use of 5 phr of Cab-O-Sil S-17 in compound -981 results in a substantial increase in compression set (see -973: +6% for cylinder and +13% for plied disk).

g. Tear Strength -- Increasing the peroxide level from 0.5 to 1.5 phr results in a decrease in tear strength from 139 to 77 ppi. The use of Tribase (-979) results in essentially no change in tear strength (both -973 and -979 ca. 100 ppi). The use of small amounts of Silanox 101 (-980) or Cab-O-Sil S-17 (-981) results in a slight lowering of tear strength. This was just the opposite of what was expected since both of these silicas are considered to be more reinforcing than Quso WR-82.

h. Abrasion Resistance -- Most of the compounds were too soft to test. However, -978 had a good Abrasive Index (88) while -981 exhibited a poor Abrasive Index (39). Evidently the use of a small amount of Cab-O-Sil S-17 does not improve abrasion resistance.

i. Low Temperature Properties -- The Gehman T_{10} values were ca. -50°C (-58°F) for all these compounds. The YMI values were all ca. -60°C (-76°F). O-Rings fabricated from these compounds are expected to function down to -60 to -70°F.

j. Resistance to Hydraulic Fluid (MIL-H-5606-C) -- All of these compounds exhibited excellent resistance to this hydraulic fluid after 240 hrs. @ 275°F, i.e., volume swell values of less than 3.0%.

In summary, the best Dicup 40C levels for the O-ring compounds appears to be in the range of 0.5 to 1.0 phr. Faster 370°F cure rates may be obtained by the use of Tribase (detrimental to 350°F stability) in place of Stan Mag ELC or by the addition of small amounts of Silanox 101. The hardness of the O-ring stocks may be increased by the use of higher peroxide levels or by the incorporation of small amounts of Cab-O-Sil S-17 (detrimental to compression set). Surprisingly, the replacement of small amounts of Quso WR-82 with corresponding amounts of Silanox 101 or Cab-O-Sil S-17 resulted in no improvement in tear or abrasion resistance.

3. <u>Mill Processing and Post Cure Studies on Quso WR-82 and FEF Black-Reinforced Compounds (Table VIII)</u>

As part of another program in our laboratories, it was found that small amounts of polysiloxanes or silicone polymers improved mill release of the phosphonitrilic fluoroelastomer. Since good mill release is necessary for O-ring production, we have incorporated the testing of silicone-containing stocks into the present contract work. Six parts of Silastic 410 were added to both a black and a silica stock. The silica stock showed reduced stickiness but did not handle nearly as well as the black stock which gave excellent release. A larger amount of silicone will probably be necessary with the silica compounds.

Table VIII illustrates the effect of Silastic 410 (6 parts) on cure and normal stress-strain properties. Times to optimum cure were increased slightly by the silicone polymer. The cured vulcanizate stress-strain properties were unaffected in the case of the black stocks and slightly improved for the silica stock. Compression set and tear strength were not adversely affected. Further testing of these stocks is in progress.

Table VIII also shows results of a study of post-cure conditions on vulcanizate properties. Our previous work usually utilized a 24 hrs./212°F post-cure which generally provided an improvement in normal stress-strain properties. It was felt that further improvements in properties and time saving could be realized by effecting the post-cures at higher temperature. However, no significient improvements were obtained in stress-strain and hardness properties. Heat-resistance and final compression set measurements must be made before a final conclusion can be obtained as to the benefits, if any, of a post cure.

III. SUMMARY OF THIRD AND FOURTH QUARTER RESULTS

A. Continuation of Second Quarter Results

1. Aged Stress-Strain

The Quso WR-82 reinforced O-ring compounds (Table VI) exhibited excellent retention of stress-strain and hardness properties after 672 hrs. @ 300°F (air), 275°F (air) and 275°F (hydraulic fluid). It is evident that O-rings fabricated from these compounds should be serviceable after exposure to the following conditions: 672 hrs. @ 275°F (air and hydraulic fluid), 672 hrs. @ 300°F (air) and 240 hrs. @ 350°F (air).

Quso WR-82 reinforced O-ring compounds containing varying levels of Dicup 40C (Table VII) also exhibit good retention of stress-strain and hardness properties after 672 hrs. @ 275°F (air and hydraulic fluid).

Different post-cure conditions on both Quso WR-82 and FEF black reinforced O-ring compounds have essentially no effect on heat resistance @ 300°F (air). In fact the samples with no post cure appear to be essentially equal in heat resistance to the ones post cured @ 212-350°F.

In view of overall mechanical properties with and without post cures there appears to be no advantage to be gained from post curing of these O-ring compounds.

2. Low Temperature Properties

Computer print-outs for Gehman tests on all O-ring compounds contained in Tables II to VIII have been filed with the original notebook data for this project. Young's Bending Modulus* of selected stocks at low temperatures have also been filed with the original data. The addition of silicone

* Young's Bending Modulus also referred to as Young's Modulus in Flexure: (ASTM D797) in other sections of this report.

rubber, Silastic 410 (6 phr), to Quso WR-82 or FEF black reinforced compounds results in a slight improvement in low temperature flexibility, i.e., lower modulus of rigidity.

3. Compression Set

Quso WR-82 reinforced O-ring compounds (Table VII) have excellent compression set after 70 hrs. @ 275°F (air). The use of Tribase in place of Stan Mag ELC (R-191,979) resulted in a significant increase in compression set. The replacement of 10 phr Quso WR-82 with the corresponding amount of Silanox 101 resulted in a slight increase in compression set (see R-191,973). Compression set is also improved by increasing the Dicup 40C level from 0.5 to 1.5 phr, i.e., increasing crosslink density.

The addition of silicone rubber (6 phr) to Quso WR-82 and FEF black-reinforced O-ring compounds (Table VIII) results in a significant improvement in the compression set properties of these compounds at 275°F. The post-cure conditions have only a slight effect on compression set, i.e., higher post-cure temperatures, 275-350°F, produce slightly lower values.

B. Summary of Second and Third Quarter Results

1. Compounding Studies to Improve Hardness and Tear Resistance of Carbon Black Reinforced O-Ring Compounds (R-193,225-227 and -218, -219) (Table IX)

Studies conducted in the first two quarters of this contract (Tables II, IV, V and VIII) revealed that carbon black reinforced O-ring compounds have relatively low hardness values (43-67) and tear strength (73°F) (27 to 111 ppi). A highly reinforcing carbon black, SAF, was evaluated with FEF and Austin black in compounds R-193,225, 226 and 227. Chem-Link 30 (3 phr), a crosslink promoter, was added in compound R-193,218 to increase modulus and hardness. Cab-O-lite P-4 (20 phr) was added to compound R-193,219 to increase hardness. The physical properties of these O-ring compounds will now be discussed in detail.

a. Monsanto Rheometer Cure -- All compounds exhibit good scorch resistance at 335°F with the exception of -218 which has a scorch time of 1.6 min. All five compounds have suitable cure characteristics at 370°F with optimum cures generally occurring at 4 to 8 minutes. It should be noted that a larger amount of peroxide must be added to achieve a good cure state when SAF is used as a reinforcing agent. Chem-Link 30 appears to function as a co-agent since it increases the cure state. Cab-O-lite P-4 appears to function as an accelerator, i.e., faster cure rate.

b. Stress-Strain Properties -- The stress-strain properties of these compounds are considered adequate for O-ring applications. However, SAF black does not appear to provide the degree of reinforcement that would be anticipated.

c. Aged Stress-Strain Properties -- These compounds have fair retention of properties after 672 hrs. @ 275°F (air) and 240 hrs. @ 300°F (air). However, after 240 hrs. @ 350°F (air) and 672 hrs. @ 275°F (hydraulic fluid -- Mil-H-5606-C) these compounds exhibit a significant loss in modulus and tensile strength. The heat resistance of these compounds may not be adequate for O-ring applications involving long term use (1000 hrs.) at 275°F in hydraulic fluids.

d. Normal and Aged Shore A Hardness -- The addition of Chem-Link 30 (-218) and Cab-O-lite P-4 (-219) did not result in an increase in hardness. The hardness values of these compounds (44-53) are considered too low for O-ring applications. These compounds have good retention of hardness upon extended aging at 275-350°F in air and at 275°F in hydraulic fluid.

e. Compression Set (70 hrs. @ 275°F) -- All compounds exhibit fair compression set except -226 which contains 10 phr of SAF black. Compound -218 exhibits the lowest compression set (25%) which is associated with its higher cure state.

f. Tear Strength (73°F) -- These compounds exhibit only fair tear strength (74-112 ppi). The use of 10 phr of SAF black (-226) evidently results in a modest increase in tear strength (112 ppi) relative to the other compounds.

g. Abrasive Index -- These compounds have fair-good abrasion resistance. Compound -219, containing Cab-O-lite P-4, exhibits the best abrasive index (100%).

h. Low Temperature Properties -- (original data on file at AMMRC) The Gehman T_{10} values range from -49 to -63°F while the apparent modulus of rigidity @ -70°F ranges from 1735 to 2994 psi. The Young's Bending Modulus at -58°F ranges from 1619 to 3245 psi. On the basis of these data, these compounds should be serviceable down to -65°F.

i. Resistance to Fluids -- All of these compounds have excellent resistance to ASTM Fuel C, i.e., volume swell ranges from 7.5 to 9.2%. All five compounds also have excellent resistance to Mil-H-5606-C hydraulic fluid at 73 and 275°F.

2. Phosphonitrilic Fluoroelastomer O-Ring Compounds Submitted to Parker Seal for Evaluation (End of Second Quarter)(Table X)

Two compounds were submitted to Parker Seal for fabrication and physical testing of O-rings. Compound R-193,228 was a Quso WR-82 reinforced compound containing Dicup 40C curing agent. Compound -229 was identical to -228 except for the addition of Silastic 410 (15 phr)(silicone rubber) to improve mill release. The results of physical testing of these compounds at Firestone will be discussed prior to discussion of Parker Seal's results.

a. Rubber Mill Processing -- Compound -228 sticks to mill rolls and tends to split to both rolls. However, this stock exhibited good green strength and formed a smooth sheet. The addition of 15 phr of Silastic 410, compound -228, results in improved mill release and less tendency to split to both rolls. Both compounds are still quite difficult to process on a rubber mill.

b. Stress-Strain -- Both compounds have stress-strain properties that are acceptable for O-ring applications, i.e., high 50 and 100% moduli, good tensile strengths and elongations (at break) of 115-143%. The addition of Silastic 410 (15 phr) results in a modest increase in the state of cure.

c. Shore A Hardness -- The hardness value for -228 (55) is slightly low for O-rings, but the value for -229 (70) is considered acceptable.

d. Compression Set (70 hrs. @ 275°F) -- Both compounds have excellent compression set properties. Compound -229, containing Silastic 410, has slightly lower compression set than -228 which may be attributed to the higher cure state of -228. The compression set of plied disks appears to be about 7-8% higher than those for cylinders. Both compounds have compression set properties suitable for O-ring applications.

e. Tear Strength -- Both compounds have fair tear strength at 73°F (ca. 110 ppi). Compounding studies should be conducted to increase tear strengths into the 150-250 ppi range.

f. NBS Abrasive Index -- The abrasive index for -228 was 110% of the control. This is considered excellent abrasion resistance since the control is a MPC black-reinforced NR vulcanizate (ASTM D-163 The abrasion resistance of -229 was not determined due to lack of a sample.

g. Aged Stress-Strain -- Stress-strain measurements were carried out on cut-ring specimens that are more similar to O-ring geometry than are dumbbell specimens. Thus, the stress-strain data obtained on cut-ring specimens should approximate data for O-ring specimens. The stress-strain properties of unaged cut-ring specimens are quite different from those of dumbbells. The cut-ring specimens have lower moduli, slightly lower tensile strength, and higher elongation at break relative to dumbbell specimens. The elongation (at break) specification for O-rings is generally stated to be 150% to allow for safety in demolding of specimens as well as installation over shafts. Therefore, cut-ring specimens should be used for stress-strain measurements on O-ring compounds. Also, a higher level of peroxide should be used than would be indicated by stress-strain measurements on dumbbell specimens.

Cut-ring specimens, both -228 and -229, have excellent retention of stress-strain properties after aging at 672 hrs. @ 275°F and 300°F (air) and 336 hrs. @ 350°F (air) and 672 hrs. @ 275°F (hydraulic fluid -- Mil-H-5606-C). The 50 and 100% moduli actually increase upon aging in air at 275-350°F. In summary, these O-ring compounds exhibit excellent heat resistance at 275°F in hydraulic fluid and 275-350°F in air.

h. Low Temperature Properties -- (Figures 7-10, Tables XXII and XXIII) Compound -228 has a T_{10} value of -58°F and an apparent modulus of rigidity at -67°F of 2937 psi. Compound -229 has a T_{10} value of -67°F and an apparent modulus of rigidity at

-67°F of 1749 psi. Therefore, the addition of Silastic 410 (15 phr) results in a modest improvement in low temperature flexibility. The Young's Bending Modulus at -58°F for -228 is 3671 psi. Both of these compounds should be flexible down to -65 to -70°F.

i. Fluid Resistance -- Compound -228 has excellent resistance to ASTM Fuel C and Mil-H-5606-C at 75°F. The addition of Silastic 410 (15 phr), compound -229, results in decreased resistance to ASTM Fuel C (20.6 % volume swell), but resistance to Mil-H-5606-C is still good (6.2% volume swell). In fact, a volume swell of 2-6% in hydraulic fluid would be expected to provide better sealing properties than the 0.5% value observed for -228. Both of these compounds are considered acceptable for use in O-rings operating in Mil-H-5606-C.

j. Parker Seal's Evaluation of Compounds R-193,228 and R-193,229 (Appendix II contains the report supplied by Parker Seal)

1. Mill Processing -- Both stocks exhibited poor mill processing, i.e., stick to mill rolls. The stock containing Silastic 410 (15 phr) had slightly better mill release but lower green strength. Additional Dicup 40C (1 phr) was added to compound R-193,228 to increase the cure state.

2. Monsanto Rheometer Cure -- Both compounds have acceptable cure rates at 370°F (6-8 minutes). Compound -228 appears to have a slightly higher cure state.

3. Stress-Strain (O-Ring Specimens) -- Compounds -228 and -229 have stress-strain properties comparable to the fluorosilicone O-ring control (L677-70). The stress-strain data for the O-ring specimen (-228) is comparable to the stress-strain data obtained on cut-ring specimens of the same compound at Firestone.

4. Shore A Hardness -- The Shore A Hardness for -228 and -229 (65 and 63, respectively) are about 10 points lower than the fluorosilicone control.

5. Resistance to Hydraulic Fluids, Mil-H-5606-C and -H-83282 -- Both compound -228 and -229 have fair resistance to Mil-H-5606-C at 302°F. Modulus and hardness exhibit fairly large decreases, tensile strength decreases slightly and elongation (@ break) increases by a large amount. The % weight change and volume swell are also fairly high. In Mil-H-83282 compounds, -228 and -229 exhibit slightly better retention of stress-strain properties. Weight change and % volume swell are also much lower.

6. Compression Set (70 hrs. @ 302°F) -- Compounds -228 and -229 have good set properties after 70 hrs. @ 302°F in air (35%). However, under the same conditions in Mil-H-5606-C the % compression set values are much higher (49-57%). These data imply that there is more degradation of these compounds in Mil-H-5606-C than in air under comparable conditions.

7. Dynamic Seal "Chew" Tests -- O-Rings constructed from compounds -228 and -229 were evaluated in Parker Seal's "Chew" tester[6]. The O-ring is mounted in a holder in contact with Mil-H-5606-C while a rod is cycled through the center of the O-ring. Both of these O-rings were compared to a fluorosilicone O-ring control (L677-70). Both O-rings (-228 and -229) failed due to rolling and tearing. This mode of failure was probably due to a combination of low modulus and low tear strength. The prime objective in future O-ring development studies should be to improve modulus and tear strength.

8. Low Temperature Performance -- It is generally accepted that an O-ring will effectively seal 10°F below its TR(10) value. The TR(10) value for -228 is -67°F and indicates service down to -77°F. The TR(10) value for -229 is -79°F and indicates service down to -89°F. The addition of Silastic 410 (15 phr) extends the lower service temperature by 12°F.

3. Phosphonitrilic Fluoroelastomer O-Ring Compound Sent to the Army (Watertown) for Environmental Testing (Table XI)

This compound (R-194,234) contained Quso WR-82 (30 phr) reinforcing silica and the standard stabilization and cure system. The masterbatch (all components except stabilizer and peroxide) was mixed in a Brabender mixer (10 batches). These masterbatches were then blended on a rubber mill and the stabilizer and peroxide were added and thoroughly mixed with the masterbatches.

a. Stress-Strain -- Measurements were made on dumbbell specimens which were cut both with and against mill grain. The specimens cut against mill grain have slightly lower moduli and tensile strengths and slightly higher elongations and tension set values. The cut-ring specimens (.050" slabs) have 50% moduli slightly lower than the dumbbell specimens which were cut against the grain. The 100% moduli are slightly higher than the against-grain dumbbell value, while tensile strength is essentially the same as the with-grain dumbbell value. The elongation at break is higher than either dumbbell value. However, when ring specimens are cut from 0.075" slabs the 50% and 100% moduli are lower than the against-grain dumbbell value. The tensile strength is again essentially the same as the with-grain dumbbell value, while the elongation (at break) is much higher than either dumbbell value. The stress-strain properties of cut-ring specimens do not appear to correlate with grain effects and must be more a result of the geometry of the specimen.

b. Shore A Hardness -- The hardness value of 65 durometer is acceptable for O-ring applications.

c. % Compression Set -- The % compression set values at 275, 300 and 325°F are 34, 38 and 39, respectively. These values are acceptable for most O-ring applications. It is significant that the compression set changes so little over a range of 275 to 325°F.

d. <u>NBS Abrasive Index</u> -- The value of 88 is considered to indicate excellent abrasion resistance.

e. <u>Aged Stress-Strain</u> -- Stress-strain measurements were carried out on aged cut-ring and dumbbell specimens. The moduli of the cut-ring specimens actually increased on aging at 300 and 350°F in air. The tensile strength is essentially unchanged after 240 hrs. at 300°F and undergoes only a slight decrease after 336 hrs. After 336 hrs. at 350°F in air the tensile strength has decreased to 597 psi. The elongation (@ break) decreases upon aging at both 300 and 350°F.

The 50% and 100% moduli of dumbbell specimens also increase upon aging at 300 and 350°F in air. The tensile strength is essentially unchanged after 336 hrs. at 300°F in air and decreases to 451 psi after 336 hrs. at 350°F in air. The elongation (@ break) also decreases upon aging at 300 and 350°F in air. Although the 50% and 100% moduli of the dumbbell specimens are originally much higher than those of the cut-ring specimens, upon aging at 350°F the values for cut-ring and dumbbell specimens gradually approach each other. This effect results in a much better % retention of stress-strain properties for cut-ring specimens relative to dumbbell specimens. The greater % loss of stress-strain properties may result from a combination of thermal degradation and loss of mill grain contribution (orientation effect). Therefore, it is concluded that phosphonitrilic fluoroelastomer O-ring compounds have better heat resistance at 300-350°F than was previously indicated by retention of with-grain dumbbell stress-strain measurements. It now appears that O-ring seals constructed from this compound would remain serviceable after 336 hrs. at 350°F.

4. Compounding Studies to Improve Cut-Ring Stress-Strain Properties (Table XII)

Stress-strain measurements were conducted on cut-ring and dumbbell specimens (with and against grain) on compounds containing varying levels of silica and peroxide (compounds R-193,235 thru -239). In compound -235 the Quso WR-82 was omitted to determine if this reinforcing agent had any effect on stress-strain measurements with and against mill grain. This vulcanized gum exhibits essentially the same stress-strain properties for dumbbell specimens cut with and against grain. However, the cut-ring specimen exhibits lower moduli, approximately the same tensile strength and higher elongation than the dumbbell specimens. Compound -236 is the same as -235 except for the addition of 20 phr of Quso WR-82. Stress-strain properties of dumbbells cut with grain now have higher moduli, higher tensile strength, lower elongation and lower tension set than specimens cut against grain. The moduli of the cut-ring specimens are lower than those of the against-grain dumbbell specimen.

Tensile strength and elongation at break fall in between the values for dumbbells cut with and against grain. Therefore, the orientation or "grain effect" observed for phosphonitrilic fluoroelastomer O-ring compounds is a direct result of the incorporation of silica reinforcing agents.

The addition of 25 and 30 phr of Quso WR-82, compounds -223 and -238, respectively, results in an increase in the 50% and 100% moduli. The tensile strength appears to reach a maximum at ca. 25 phr while the elongation (@ break) remains essentially invariant. The "grain effect" is also apparent in these compounds. The stress-strain properties of the cut-ring specimen appears to correlate better with those of the against-grain dumbbell specimen.

Compound -239 was identical to -238 except for the addition of more peroxide (1 phr) to achieve a higher cure state. There appears to be less differences between stress-strain properties on dumbbell specimens cut with and against grain on this highly cured sample. However, the cut-ring specimen still exhibits a much lower 50% modulus relative to the dumbbell specimens. The elongations (@ break) of all three specimens are essentially the same.

Shore A Hardness for compounds -235 and -236 are too low for most O-ring applications. However, the values for -237, -238 and -239 are considered suitable since they are in the 65 to 75 durometer range. On the basis of cut-ring stress-strain data, compound -238 appears to be the best O-ring compound.

5. Evaluation of Quso G-32 Silica Treated with a Silane Coupling Agent (Union Carbide A-174)(Table XIII)

Quso G-32 is a precipitated silica with the following properties:

(1.) Ultimate Particle Size (millimicron) - 13

(2.) Surface Area (sq. m./g.) - 300

(3.) pH - 8.5

This silica was treated with Union Carbide Silane A-174, gamma-methacryloxypropyltrimethoxysilane in methanol. This silane coated silica was compared to the untreated silica by compounding into standard formulations -- R-193,255 (control), R-193,256 and R-193,257. Silane treatment of this silica appears to improve the % tension set of the reinforced vulcanizate. However, no improvement occurred in stress-strain, compression set, tear strength, abrasion resistance, heat resistance and low temperature properties.

6. Compounding Studies to Improve Stress-Strain Properties of Cut-Ring Specimens (Table XIV)

Phosphonitrilic fluoroelastomer O-ring stocks were compounded with Quso WR-82, FEF black and Cab-O-lite P-4 and a relatively high level of peroxide to generate a high cure state. Cab-O-lite P-4 is a relatively low surface area (2.2 m^2/g) calcium metasilicate with a highly basic surface (pH - 9.9). This semi-reinforcing filler was added to increase the hardness of both Quso WR and FEF black reinforced O-ring compounds.

a. Mill Processing -- All compounds exhibited poor mill processing, i.e., low green strength, stick to rolls and splitting to both rolls.

b. Monsanto Rheometer Cure -- All compounds were relatively scorchy @ 335°F. The optimum cure times @ 370°F ranged from 2.7 to 9.5 minutes and are considered acceptable factory cure cycles. Cab-O-lite P-4 acts as an accelerator in the Quso WR-82 reinforced compound (R-193,262) but not in the FEF black reinforced compound. The FEF black reinforced compounds exhibit higher cure rates @ 370°F than do the Quso WR-82 reinforced stocks. Compound -264, containing 50 phr of Cab-O-lite P-4, exhibits an extremely high cure rate @ 370°F.

c. Stress-Strain Properties -- Both the Quso WR-82 and FEF black reinforced compounds have good moduli, tensile strengths and elongations. The addition of Cab-O-lite P-4 (30 phr) to both Quso WR-82 and FEF black reinforced compounds results in a very slight improvement in low strain modulus (50%) but a modest loss in high strain modulus (100%) and tensile strength.

d. Shore A Hardness -- All compounds except -264 (50 phr Cab-O-lite P-4) have hardness values acceptable for O-ring applications. The addition of Cab-O-lite P-4 (30 phr) results in a modest increase (+8-10 points) in hardness of both silica and black reinforced compounds.

e. Compression Set -- The silica reinforced compounds (-259, -260) have excellent compression set resistance after 70 hrs. @ 275 and 300°F. After 138 hrs. @ 350°F the compression set (%) is still only 48--a very respectable value under these severe conditions. The compression set resistance of the black reinforced compound (-261) is slightly lower than the silica reinforced compounds @ 275-300°F and much lower @350°F. The addition of Cab-O-lite P-4 (30 phr) to either silica or black reinforced compounds results in a modest decrease in compression set resistance. Compound -264, containing Cab-O-lite P-4 (50 phr) has the best compression set resistance at 275-300°F but is not as good as the silica reinforced compounds @ 350°F. In general, the compression set properties of all of these compounds are considered adequate for O-ring applications.

f. Tear Strength (73°F) -- The FEF black reinforced compound (-261) exhibits excellent tear resistance (243 ppi). The Quso WR-82 compounds (-259 and -260) also have good tear strength, 161 and 204 ppi, respectively. There is a modest increase in tear strength (+43 ppi) in progressing from 25 to 30 phr of Quso WR-82. The addition of Cab-O-lite P-4 (30 phr) results in modest decreases in tear resistance. Compound -264, containing Cab-O-lite P-4 (50 phr) has relatively low tear strength (87 ppi). All compounds except -264 have tear resistance acceptable for O-ring applications.

g. NBS Abrasion Resistance -- The FEF black reinforced compound (-261) exhibits the best abrasion resistance (57% of the control). The abrasive indices for the remaining stocks are quite poor (26-33% of the control). The abrasion resistance of -261 is considered adequate for dynamic O-ring applications but those of the remaining compounds would be considered marginal.

h. Aged Stress-Strain Properties -- The Quso WR-82 reinforced compounds (-259 and -260) have excellent retention of stress-strain properties after 1000 hrs. @ 275°F and 300°F in air and after 240 hrs. @ 350°F in air. These compounds are highly degraded after 240 hrs. @ 400°F in air. After 1000 hrs. @ 275°F in hydraulic fluid (Mil-H-5606-C) these compounds still exhibit excellent stress-strain properties. The FEF black reinforced compound (-261) exhibits good retention of stress-strain properties after 1000 hrs. @ 275°F and 300°F in air and fair retention after 240 hrs. @ 350°F (air). This compound was also highly degraded after 240 hrs. @ 400°F in air. Compound -261 has relatively poor retention of stress-strain properties after 1000 hrs. @ 275°F in hydraulic fluid (Mil-H-5606-C). The addition of Cab-O-lite P-4 (30 phr) to both silica and black reinforced compounds results in essentially no change in heat-resistance. Compound -264, containing Cab-O-lite P-4 (50 phr) also exhibits excellent heat resistance. From the preceding data it appears that Quso WR-82 and Cab-O-lite P-4 reinforced compounds have the following approximate service lives:

1000 hrs. @ 275, 300°F in air

1000 hrs. @ 275°F in hydraulic fluid (Mil-H-5606-C)

240 hrs. @ 400°F in air

i. Low Temperature Properties -- Plots of Gehman twist angle and apparent modulus of rigidity (G) versus temperature are on file with the original data at AMMRC. The apparent modulus of rigidity values @ -70°F indicate that these compounds should be serviceable at this temperature. The Young's Bending Modulus values also indicate good flexibility @ -70°F.

7. Evaluation of Selected Vulcanization Agents for Phosphonitrilic Fluoroelastomer O-Ring Compounds (Tables XV and XVI)

Selected organic peroxides were evaluated in a standard O-ring formulation -- Polymer - 100, Quso WR-82 - 30, Stan Mag ELC - 6, $(8\text{-HQ})_2Zn$ - 2. A detailed description of the peroxides used in this study are summarized in the glossary. All peroxides were evaluated at the same RO· generating level so that relative vulcanizing efficiencies could be correlated.

The use of Dicup R (0.8 phr) in compound R-193,265 resulted in approximately the same cure characteristics, cure state, stress-strain, hardness, % compression set and heat resistance as obtained with Dicup 40C (2.0 phr) in -266. Evidently the presence of 1.2 phr of calcium carbonate in Dicup 40C has no effect on cure, overall mechanical properties and heat resistance. Stan Mag ELC (6 phr) (magnesium oxide) was omitted in compound -267. When compared to the control (-266) it is obvious that Stan Mag ELC has no effect on cure rate but does result in a slightly higher cure state. This magnesium oxide appears to have no effect on heat resistance at 300 and 350°F. The hardness is slightly lower for the compound without the magnesium oxide and the compression set is slightly lower. The only function of magnesium oxide appears to be that of a mild crosslinking agent. It is possible that the use of a slightly higher level of peroxide would accomplish the same effect.

Cadox BS (benzoyl peroxide) was added at a level of 1.44 phr in compound -268. This peroxide did not provide a good cure state at 250°F. Cadox TS-50, 2,4-dichlorobenzoyl peroxide, was tested at a level of 1.84 phr in compound -269. This peroxide also proved to be an extremely poor curing agent.

Vulcup R, α,α'-bis(t-butylperoxy)diisopropylbenzene, was evaluated at a level of 0.5 phr in compound R-193,270. This peroxide provides a much slower cure than Dicup R at 340°F, optimum cure of 22 versus 10 minutes. However, Vulcup R appears to be considerably more efficient than Dicup 40C on the basis of rheometer cure and stress-strain data. This peroxide also provides higher hardness, lower compression set and comparable heat resistance at 300 and 350°F. Another advantage of Vulcup R is that no odor is imparted to the cured specimens as is the case in Dicup R cured samples (acetophenone odor). Therefore, it is not necessary to post cure specimens cured with Vulcup R to eliminate offensive odors. It is highly recommended that Dicup R or 40C should be replaced by Vulcup R.

Varox, 2,5-bis(tert-butyl peroxy)-2,5-dimethylhexane, (50% active) was evaluated at a level of 0.86 phr in compound R-193,271. This peroxide exhibits a much slower cure rate at 340°F than Dicup R, optimum cure of 39.5 versus 10.0 minutes. This peroxide is less efficient than Dicup R as evidenced by rheometer, stress-strain and hardness properties. The compression set resistance appears to be slightly better considering the modestly lower cure state. The heat resistance at 300 and 350°F appears to be comparable to the Dicup R cured compound.

Percadox 29140, Luperco 130 XL, Luperco 230 XL and di-t-butyl peroxide were evaluated in compounds R-193,272, -273, -274 and -275, respectively. These peroxides were less efficient than Dicup R, Vulcup R or Varox and generally resulted in poorer mechanical properties and heat resistance. In summary, Vulcup R is the recommended curing agent for phosphonitrilic fluoroelastomer O-ring compounds. Dicup R, Dicup 40C and Varox are also considered acceptable curing agents.

8. Dumbbell Versus Cut-Ring Stress-Strain Measurements -- Correlations Between Testing at Firestone and Horizons Research, Inc. (Tables XVII and XVII-A)

Previous studies have shown that a substantial difference exists between cut-ring and dumbbell stress-strain data obtained on phosphonitrilic fluoroelastomer stocks tested at Firestone. The cut-ring specimens exhibit lower moduli at 50 and 100% strain, higher elongation at break and slightly lower tensile strength relative to dumbbell specimens.

Furthermore, cut-ring stress-strain measurements conducted at Firestone did not show good agreement with tests made by Horizons Research on the same stock. i.e., Firestone tests showed lower 50 and 100% moduli, higher elongations at break and essentially the same tensile strength.

In an effort to uncover the factors causing these anomalies a "round-robin" testing program was conducted between Firestone and Horizons Research. The phosphonitrilic fluoroelastomer O-ring stock (R-193,276) selected for this study is described in Table XVII. Four 6" x 6" x 0.075" slabs were cured at the same time in a press for 60 minutes at 320°F followed by a post cure of 4 hours at 350°F in a forced-air oven. A complete description of the method of testing and stress-strain results is given in Table XVII-A. All of the specimens were cut and tested on the same day to eliminate any effects due to sample aging. The conclusions from these tests are as follows:

a. The differences between cut-ring and dumbbell stress-strain data were traced to a malfunction in the computer read-out of the Instron. This malfunction occurs only for stress-strain curves with extremely steep slopes which are characterisitc of highly cured phosphonitrilic fluoroelastomer O-ring stocks which have high low strain moduli and low elongations ($< 150\%$). A correction has been made in the computer read-out circuit and it now appears that this problem has been solved. Ring tensile data from a computer print-out are being compared to data calculated from the Instron chart for a large number of samples.

b. If calculations are made from the Instron charts, cut-ring stress-strain compares fairly well with dumbbell data and also to cut-ring stress-strain measured at Horizons Research. However, there appears to be a substantial difference between cut-ring and dumbbell data for slab No. 2. These differences may result from a small contribution from mill grain or uneven dispersion.

c. The dispersion of this phosphonitrilic fluoroelastomer O-ring stock appears to be quite good in the same slab and also between slabs, i.e., standard deviations are quite small. However, there appears to be some differences in cure state between the 4 slabs, i.e., slabs 2 and 3 exhibit higher moduli than 1 and 4.

d. With regard to precision, the following conclusions are based on standard deviations:

   1. Ring Cutting -- Firestone is slightly better.
   2. Ring Testing -- Firestone is slightly better.

However, the average values of stress-strain data between Firestone and Horizons compare very well.

In summary, most of the disagreements between ring and dumbbell tensile have been eliminated by either calculating the data from Instron charts or by modifying the computer read-out circuit. The agreement between Horizons and Firestone cut-ring stress-strain data is now considered excellent. All future stress-strain measurements on phosphonitrilic fluoroelastomer compounds will be made on cut-ring specimens.

IV. EXPERIMENTAL

A. Mixing and Processing - ASTM D-15

All phosphonitrilic fluoroelastomers were first masterbatched in a Brabender mixer (Plasticorder PLV-150). The silica or carbon black was added to the polymer in the Brabender and mixed for five minutes. The magnesium oxide (Stan Mag ELC) was then added and the mixing was continued for another five minutes. The masterbatch was dumped and added to a small rubber mill (3" x 6"). The stabilizer and peroxide curing agents were then added to the masterbatch and mixed for ten minutes. The maximum temperature of the batch was maintained below 160°F. The finished compounds were then sheeted out to the desired thickness on a 10" x 20" mill. The temperature of the rolls should be maintained at 130 ± 10°F for best processing. In general, phosphonitrilic fluoroelastomer O-ring compounds exhibit relatively poor processing on a rubber mill, i.e., stick to mill rolls and have low green strength.

B. Physical Testing in general was conducted according to ASTM specifications unless specified otherwise.

1. Williams Plasticity -- ASTM D-926.

2. Mooney Viscosity -- ASTM D-1417 -- Scott STI/200 Mooney viscometer.

3. Specific Gravity was measured on small cylinders of polymer.

4. Cure Properties -- ASTM D-2084 -- Monsanto Rheometer, Model 100 -- Mini Die, 100 rpm, 1° arc.

5. Stress-Strain -- An Instron 1130 was used for all measurements.

   a. Dumbbell -- ASTM D-412.

   b. Cut-Ring -- ASTM D-412.

   c. O-Ring -- ASTM D-1414.

   d. Aged in Air -- ASTM D-5733 -- Specimens were contained for the specified times in forced-air ovens maintained at 275, 300, 350 or 400°F.

   e. Aged in Fluids -- ASTM D-1460 -- An aluminum block-test tube aging apparatus, Scott Model L.G., was employed for these measurements.

6. Shore A Hardness -- ASTM D-2240 -- Measurements were made on molded cylinders (0.250" x 0.530" diameter).

   a. Aged in Air -- ASTM D-573 -- forced-air ovens.

   b. Aged in Fluids -- ASTM D-1460 -- An aluminum block-test tube aging apparatus, Scott Model L.G., was used for these measurements.

7. Compression Set -- ASTM D-359, Method B, 25% Deflection -- A molded cylinder (0.500" x 1.13" diameter) was used in these tests. Measurements were also made on plied (0.500" x 0.075") disks in specific cases.

8. Tear Strength -- ASTM D-624, Die B, specimens were cut-out of 6" x 6" x 0.075" slabs. Specimens were nicked across the specimen at the center of the inner concave edge.

9. Abrasion Resistance -- ASTM D-1630 -- Tests were conducted on Young's Modulus blocks.

10. Young's Bending Modulus -- ASTM D-797.

11. Gehman Low Temperature Torsion -- ASTM D-1053 -- Specimens were cut-out of 6" x 6" x 0.075" slabs. A Wallace apparatus (Model L.15) was used for testing specimens immersed in isooctane. Dry Ice was added until the temperature decreased to -80°F, then the temperature was slowly increased to the desired test temperature by a small immersion heater. Measurements in nitrogen (gas) were made on an instrument constructed by Firestone Synthetic Rubber and Latex Company.

12. Extrusion Properties -- ASTM D-2230 -- Measurements were made by use of a Brabender extruder.

13. Fluid Resistance -- % Weight Change, % Volume Swell, % Extracted -- ASTM D-1460.

14. O-Ring Specimens -- ASTM D-1414.

15. Temperature Retraction Test -- ASTM D-1329 -- The samples are elongated at a given strain and frozen (-70°C). The temperature is then slowly increased (1°C/min.) and the percent retraction is measured. The temperature at which the specimen retracts 10% is designated TR 10. Parker Seal has stated that a seal should function at 10-15°F below the TR 10 value.

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APPENDICES

I. GLOSSARY

II. PARKER SEAL REPORTS

III. TABLES

IV. FIGURES

GLOSSARY

Compound Ingredient	Chemical Name/Description	Supplier
AC Polyethylene	Polyethylene	Allied Chemical
Austin Black	Coal Fines	Slab Fork Coal Co.
Cab-O-lite P-4	Calcium Metasilicate	Interpace Corp.
Cab-O-Sil S-17	Fumed Silica	Cabot Corp.
Cadox BS	Benzoyl Peroxide (50% active)	Noury Chem. Corp.
Cadox TS-50	2,4-Dichlorobenzoyl Peroxide (50% active)	Noury Chem. Corp.
Chem-Link 30	Coagent/Trimethylol Propane Trimethacrylate	Ware Chem. Corp.
Dicup 40C	Dicumyl Peroxide (40% active)	Hercules
Dicup R	Dicumyl Peroxide (96-100% active)	Hercules
Di-t-Butyl Peroxide	Di-t-Butyl Peroxide	Pennwalt Lucidol Chemicals
Epolene C-305-G	Polyolefin Wax	Harwick Chem. Co.
FEF Black	Fast Extrusion Furnace -- Carbon Black	Several Suppliers
Graphite	Superior 5033X	Superior Graphite Co.
Mag Carb L	Magnesium Carbonate	C.P. Hall Co.
Mistron Vapor	Magnesium Silicate	Cyprus Mines, United Serra Div.
MT Black	Medium Thermal -- Carbon Black	Several Suppliers

Compound Ingredient	Chemical Name/Description	Supplier
Nulok 321-L	Amino Silane Coated Clay	Huber
Percadox 29/40	Peroxide	Noury Chem. Corp.
Quso WR-82	Silane Coated, Precipitated Silica	Philadelphia Quartz Co.
Quso G-32	Precipitated Silica	Philadelphia Quartz Co.
SAF Black	Super Abrasion Furnace -- Carbon Black	Several Suppliers
Silanox 101	Silane Coated-Fumed Silica (Now "Tullanox")	Cabot Corp. (Tulco Inc.)
Silastic 410	Poly(dimethyl)siloxane (Vinyl Cure Site)	Dow Corning
Silastic 430	Poly(dimethyl)siloxane (Vinyl Cure Site)	Dow Corning
Stabilizer (Against Heat)	Bis-(8-Hydroxyquinolate) Zinc (II)	Ashland Chem., Fine Chemicals Dept.
Stan Mag ELC	High Activity Magnesium Oxide	Harwick Chem. Co.
Teflon 6	Fibrous Teflon	DuPont
Tribase	Hydrous Tribasic Lead Sulfate	National Lead Co.
Union Carbide Silane A-151	Vinyltriethoxysilane	Union Carbide Corp.
Union Carbide Silane A-174	Gamma-Methacryloxypropyl-trimethoxy Silane	Union Carbide Corp.

Compound Ingredient	Chemical Name/Description	Supplier
Union Carbide Silane A-1100	Gamma-Aminopropyltri-methoxy Silane	Union Carbide Corp.
Varox	2,5 bis(t-butyl peroxy)-2,5-dimethylhexane (50% active)	R. T. Vanderbilt Co., Inc.
Vulcup R	α,α'-bis(t-butyl peroxy)-diisopropylbenzene	Hercules
Luperco 130 XL	2,5-Dimethyl-2,5-bis(t-butyl-peroxy) Hexyne-3 (45% active)	Pennwalt, Lucidol Chemicals
Luperco 230 XL	n-Butyl-4,4-bis(t-butyl-peroxy) Valerate (50% active)	Pennwalt, Lucidol Chemicals

APPENDIX II-A

STATUS REPORT I -- FIRESTONE PHOSPHAZENE

BACKGROUND:

As per our agreement with Firestone to evaluate their phosphonitrilic compounds, Parker Seal received June 5, 1974 one pound samples of two phosphonitrilic fluoroelastomer stocks. These were labeled R-191,959 (nonblack) and R-191,941 (black). These materials were Firestone's best effort to date with the phosphonitrilic polymer.

PROCESSING CONCLUSIONS:

Both stocks processed on the mill with difficulty. Handling properties were quite similar to the poorer processing fluorosilicone polymers such as L449-65. Severe sticking was encountered with the nonblack, and to a lesser degree with the black stock. The problem is a smearing and subsequent adhesion to the mill rolls. It had been suggested this might be alleviated by the addition of small amounts of silicone polymer. A small side experiment with a previous phosphonitrilic fluoroelastomer compound proved this to be true at a level of 15 pphr. Although the effect on physical properties was not determined, the processing problem was eliminated.

RHEOMETRY AND VULCANIZED PROPERTIES:

Monsanto Rheometer curves were run on each compound to determine optimum cure times at 370°F. The results were:

	90% Optimum Cure	Torque/IPS
R-191,959 (nonblack)	5 minutes	61
R-191,941 (black)	7 minutes	40

(The final torque values were indicative of the compounds resultant modulus @ 100% elongation.) In addition to the optimum cure time a 24 hour @ 302°F post cure was also used to remove any residual peroxide.

The parts produced were 2-214 O-rings (.139" c.s.). The original physicals were as follows:

	R-191,941 (black)	R-191,959 (nonblack)	L677-70
Hardness, Shore A	60	60	74
Tensile Strength (psi)	762	764	1130
Elongation (%)	162	119	165
Modulus @ 100% E (psi)	455	576	665

As can be seen, both stocks are still weak in comparison to a good fluorosilicone. Apparently there is still a lack of good polymer-filler adhesion in these compounds.

AGING AND DYNAMIC PROPERTIES:

The following aging and low temperature values were obtained:

	R-191,959	R-191,941
Fluid Age in MIL-H-5606 70 Hours @ 302°F		
Hardness, (Change, pts)	56(-4)	52(+8)
Tensile Strength (Change, %)	986(+29)	426(-17)
Elongation, (Change, %)	161(+35)	222(+37)
Modulus @ 100% (Change, %)	440(-24)	238(-49)
Volume Change, %	+4.7	+6.2
Weight Change, %	+1.8	+3.1
Fluid Age in MIL-H-83282 70 Hours @ 302°F		
Hardness, (Change, pts)	57(-3)	56(-4)
Tensile Strength, (Change, %)	910(+19)	604(-21)
Elongation, (Change, %)	143(+20)	210(+30)
Modulus @ 100%, (Change, %)	478(-17)	269(-42)
Volume Change, %	+1.4	+1.8
Weight Change, %	+0.4	+1.2
Compression Set (5) 70 Hours @ 302°F	22.5	48.1
TR-10	-71°F	-69°F

Both compounds were run on our dynamic seal "chew" tester against L677-70 (fluorosilicone) in MIL-H-5606 fluid. It was in this test a glimmer of improvement over previous phosphonitrilic fluoroelastomer compounds appeared. Leakage and seal damage was approximately equivalent to that of the fluorosilicone in the nonblack stock. It is believed that the improved set contributed significantly to this result.

RECOMMENDATIONS:

1. The processing could very possibly be improved via the addition of small amounts of silicone like material. Its effect on physical properties should then be explored.

2. The overall hardness of the stock must be improved, either through the use of additional fillers or via a crosslinking additive.

3. Compression set requires still further improvement in order to retain its sealing ability over time and temperature.

4. Abrasion resistance in these compounds has been shown to be at least equal to fluorosilicone, but in the final formula it must significantly be improved on if phosphonitrilic fluoroelastomer is to become a viable dynamic seal.

APPENDIX II-B

STATUS REPORT II -- FIRESTONE PHOSPHAZENE

BACKGROUND:

Parker Seal received on September 18, 1974 one pound samples of two phosphonitrilic fluoroelastomer stocks. These were labeled R-193,228 and R-193,229 and were stated to be identical except for an addition of 15 pphr of Silastic 410 as a release agent in the latter.

PROCESSING CONCLUSIONS:

The addition of 15 pphr of silicone did not markedly improve the processing of this compound. The loss in green strength created by the silicone was not adequately balanced by its release. Smearing and adhesion to the mill remain problems with both these stocks.

RHEOMETRY AND VULCANIZED PROPERTIES:

Oscillating disc Monsanto Rheometer curves were run at 3° arc at 370°F. The results were:

	90% Optimum Cure	Torque/IPS
XZ2046-10 (R-193,228 + 1 pphr Dicup)	6 minutes	30
XZ2046-11 (R-193,229)	8 minutes	25

The test parts produced were 2-214 O-rings (.139" c.s.). In addition to an optimum cure @ 370°F, all parts were given a 24 hour @ 302°F post cure.

The original physicals were as follows:

	XZ2046-10	XZ2046-11	L677-70
Hardness, Shore A	65	63	74
Tensile Strength (psi)	1020	855	1130
Elongation (%)	125	170	165
Modulus @ 100% E (psi)	737	511	665

AGING AND DYNAMIC PROPERTIES:

The following aging and low temperature values were obtained:

	XZ2046-10	XZ2046-11
Fluid Age in MIL-H-5606 70 Hours @ 302°F		
Hardness, (Change, pts)	56(-9)	58(-5)
Tensile Strength, (Change, %)	974(-5)	751(-12)
Elongation, (Change, %)	187(+50)	197(+16)
Modulus @ 100%, (Change, %)	360(-51)	326(-36)
Volume Change, %	+15.0	+10.0
Weight Change, %	+ 6.7	+ 4.6
Fluid Age in MIL-H-83282 70 Hours @ 302°F		
Hardness, (Change, pts)	57(-8)	57(-6)
Tensile Strength, (Change, %)	1040(+2)	807(-6)
Elongation, (Change, %)	160(+28)	214(+26)
Modulus @ 100%, (Change, %)	457(-38)	317(-38)
Volume Change, %	+ 2.9	+ 5.9
Weight Change, %	+ 0.8	+ 2.6
Compression Set (%) 70 Hours @ 302°F	35.4	35.3
Compression Set in MIL-H-5606 (%) 70 Hours @ 302°F	56.9	49.0
TR10	-67°F	-79°F

Both compounds were compared on our dynamic seal "chew" tester against a good fluorosilicone (Parker L677-70) in MIL-H-5606 fluid. These particular compounds proved to be less an improvement than the previous compounds that were tested. In all cases, the phosphonitrilic fluoroelastomer stocks' modes of failure, rolling and tearing, were due to mechanical instability and low tear strength. Both these problems have their roots in a lack of vulcanizate modulus.

RECOMMENDATIONS:

1. The primary problem of compound modulus improvement should be strongly emphasized. Better polymer-filler interaction is a must if phosphonitrilic fluoroelastomer is ever to be an advantage over fluorosilicone stocks.

2. Processing problems are of strictly secondary importance at this point in development. The technique of using a silicone polymer as a release agent, however, is still viable and should continue to be investigated. The investigation should especially include the newer high strength, high tear silicone polymers.

APPENDIX II-C

FINAL REPORT

FIRESTONE PHOSPHAZENE DEVELOPMENT

Firestone Comments on Parker's Final Report

The compounds shown in Table XVIII were submitted to Parker for evaluation during the final quarter of the contract period. The use of the silane coupling agents (Compounds R-194,844 to 846) was based on an earlier recommendation by Parker that coupling agents be evaluated in an effort to increase the interaction between the polymer and the filler. It was believed that this would improve both static and dynamic physical properties of these compounds.

Compound R-194,847 featured the addition of Teflon 6 and Silastic 430 to improve the processing of phosphonitrilic fluoroelastomer compounds. As detailed in the Parker report which follows, the desired reduction in smearing on the mill and improved green strength were realized, but some reduction in seal performance resulted, probably due to poor dispersion of the Teflon 6 in the compound.

Parker has noted that these four compounds represent "quantum" improvements over previously tested phosphonitrilic fluoroelastomer compounds. Their conclusion is that although further development of phosphonitrilic fluoroelastomer compounds will be required to realize their full potential in O-ring hydraulic seals, they are fast approaching fluorosilicones and in some areas they now surpass fluorosilicones. It is important to note that the phosphonitrilic fluoroelastomer O-ring seals are superior to the fluorosilicone O-ring seals with respect to extrusion resistance and abrasion resistance.

PARKER HANNIFIN

SEAL GROUP: 10567 JEFFERSON BOULEVARD, CULVER CITY, CALIFORNIA 90230, TELEPHONE (213) 837-5101

FINAL REPORT
FIRESTONE PHOSPHAZENE DEVELOPMENT

April 11, 1975

OBJECT

The purpose of this program was the development of a phosphonitrilic fluoroelastomer (PNF) based rubber compound for use in O-rings as a competitor to the fluorosilicone class of polymers. The processing characteristics of the raw compound were to be evaluated on standard rubber processing equipment. Accelerated fluid aging and wear testing were to be performed on O-ring test specimens. Recommendations were to be made concerning further improvement of PNF compounds for use in O-rings.

In this, the final phase of the program, four compounds were received and evaluated as O-ring stocks. These were R194,844 through 194,847 (see page 4.) and were relabeled with the Parker Seal numbers XN2046-12, 13, 14, and 15 respectively.

PROCESSING CONCLUSIONS

Each of the first three compounds processed poorly, much as have most of the PNF compounds developed to date. Processing on a double roll mill was difficult although a slight improvement in release was detected over previous compounds. Smearing and subsequent adhesion to the rolls persisted as the processing nemesis of these PNF materials. XN2046-15, however, represented an abrupt departure from all the compounds seen heretofore. Its processing was marked by good green strength and excellent release properties. It processed not unlike a high strength silicone and as such represented a goal obtained in PNF processing. Such a material could be readily processed as an O-ring stock with conventional methods. Unfortunately, certain trade-offs were made in fluid resistance to obtain this as will be discussed later.

MOLDING PROPERTIES

The test specimens produced were 2-214 O-rings (.139" cross section, 1" ID.) It should be noted some difficulty was encountered in the molding of -15. The stock had a slight tendency to form knit lines where it had flowed together. (The cause of this problem is suspected to be the five parts of Teflon 6 in the compound.) The thin flash of all the PNF compounds tends to be sticky and could present an obstacle to deflashing of parts in a full production set up. Each of the compounds was found to vulcanize to an optimum state of cure at 370°F after five minutes, well within the limitations of O-ring production.

VULCANIZED PROPERTIES

For ease of installation on piston rings, most O-ring seals require sufficient strength to be stretched over the piston without breaking. Experience has shown that a tensile of 1100-1200 psi and an elongation of 125% to be the approximate minimums to accomplish this task. The data show that of all the compounds, including the representative fluorosilicones, only XN2046-15 meets these requirements. (It does so at a cost to overall performance as explained below.) However, all of the PNF compounds here are quantum improvements over previous compounds in this area.

The Shore A hardness of these stocks is still somewhat low for use in the full range of dynamic applications. The optimum hardness should be about 75 Shore A to make a good dynamic ring, whereas the PNF rings continue to run below 70 without sacrifice to elongation.

In the area of modulus, the O-rings tested were also much improved, yielding 800-900 psi at 100% elongation as opposed to the 400-500 psi seen previously. This improvement in turn led to better results in the areas of extrusion and dynamic chew testing. Similarly, the hot stress-strain results confirmed this modulus improvement over a high temperature range.

EXTRUSION RESISTANCE

O-ring extrusion resistance is tested by measuring the pressure required to extrude the O-ring test specimen from a groove with diametrical clearance of .015". By performing this test at various temperatures, an excellent measure of relative extrusion resistance is produced. The results of this testing proved predictable from the modulus figures. The -12 and -14 were highest, the -13 and -15 were a second grouping, and the fluorosilicones trailed the field. This effect was also demonstrated in the chew testing.

AIR AND FLUID TESTING

In air aging, excessive hardness increases or elongation losses are to be avoided. The data show the PNF stocks to have performed reasonably well here in comparison to the fluorosilicones. It is desirable, although, to have an aged elongation above 100% and this would have been the case with the PNF if our initial elongations had been higher with the -12, -13, and -14 as were -15 and the fluorosilicone. The -15, with its small amount of silicone, proved superior over the other PNF compounds in retention of overall good properties in heat age.

In general, the results of the fluid aging proved the superiority of PNF solvent resistance over fluorosilicones regardless of the fluids in which they were tested. Swelling and general degradation was less with all the PNF stocks than the fluorosilicones. The oil aging at 350°F in the MIL-H-83282 fluid only moderately effected the PNF's, whereas the fluorosilicones were destroyed. The steam testing demonstrated well the hydrolytic stability of PNF versus fluorosilicone. The PNF compounds were so good in the area of solvent resistance that it worked to their detriment in slightly higher compression sets than would otherwise be possible with more positive swell.

The oil aging results also shed light on a peculiar defect of XN2046-15. All of the specimens tested in oil gave an uneven or lumpy swell. This was later determined to be caused by a nondispersed component, Teflon 6. The effect of the uneven swell produced high variability in the fluid age results. In the dynamic testing this swell led to actual damage of the O-ring.

Compression set is the most critical property an O-ring must possess and it is the most common downfall of experimental compounds. These phosphazenes are much improved over past compounds in this area; fast becoming comparable to at least one of the fluorosilicones and actually showed superiority in the MIL-H-83282 fluid. This conclusion was borne out in the dynamic testing.

DYNAMIC TESTING

Of all our physical tests, the rod chew tester is the closest to an actual sealing application. The test was run on all stocks in both MIL-H-5606 and MIL-H-83282 fluids. The results provided definitive proof of the PNF stocks' ability as a dynamic seal. With the exception of the -15 with its dispersion problem, the PNF compounds performed far better than either fluorosilicone. Leakage was higher in the phosphazenes than the fluorosilicones but more importantly the phosphazene took only a slight compression set while both fluorosilicones had undergone severe physical damage. It was concluded that had the test continued, both the fluorosilicones would catastrophically fail while the PNF would tend to higher leakage due to compression set. (This latter type of failure is more desirable than total failure.)

RECOMMENDATIONS

1. The initial processing problem of mill smearing is apparently solvable with the addition of a small amount of silicone. For release, the Teflon 6 could still be utilized if an adequate method of dispersion is found.

2. Compression set could still stand further improvement in order for PNF to retain its sealing ability over time and temperature.

3. A final compound incorporating the above should be given a pilot study and subsequent trial in an actual application

ORIGINAL PHYSICALS

	L449	L677	XN2046-12	XN2046-13	XN2046-14	XN2046-15
Original Physical Properties						
Hardness, Shore A, pts	57	63	67	63	72	73*
Tensile Strength, psi	692	1050	983	1040	1000	940
Elongation, %	153	245	92	114	88	121
Modulus @ 100%, psi	353	380	-	810	-	789
Specific Gravity	1.41					

AIR AND FLUID AGINGS

	L449	L677	XN2046-12	XN2046-13	XN2046-14	XN2046-15
Air Aging 70 Hours@ 302°F						
Hardness, Shore A, (Chg, pts)	58(+1)	64(+1)	70(+3)	71(+8)	79(+7)	70(-3)*
Tensile Strength, psi (Chg, %)	600(-13)	1020(-3)	1110(+13)	1120(-8)	1080(+8)	1100(+17)
Elongation,% (Chg, %)	137(-10)	230(-6)	93(+1)	73(-18)	87(-1)	159(+31)
Modulus @ 100%, (Chg, %)	362(-3)	404(+6)	-	-	-	740(-6)
Weight Change, %	-0.3	-0.4	-5.5	-5.5	-5.9	-1.3
Compression Set % of Original Deflection	29.4	16.6	24.2	49.0	35.3	31.4
Compression Set in Air @ 347°F	47.1	30.4	48.5	70.6	52.9	54.9
Fluid Age in MIL-H-5606 70 Hours @ 302°F						
Hardness, Shore A, pts	48(-9)	59(-4)	67(0)	65(-2)	72(0)	58(-15)*
Tensile Strength, psi (Chg, %)	690(0)	798(-24)	1120(+14)	1106(+6)	1210(+21)	1080(+15)
Elongation, %, (Chg, %)	213(+39)	207(-16)	109(+18)	114(0)	109(+24)	236(+95)
Modulus @ 100% (Chg, %)	210(-41)	319(-16)	1000	890(+10)	1090	454(-42)
Volume Change, %	+9.7	+7.8	+1.7	0	-1.0	+11.5
Weight Change, %	+5.9	+4.3	-0.4	-1.8	-2.7	+ 5.7
Compression Set % of Original Deflection	10.8	21.5	38.2	55.9	47.1	46.1
Fluid Age in MIL-H-5606 70 Hours @ 347°F						
Hardness, Shore A, pts	43(-14)	57(-6)	60(-7)	60(-3)	65(-7)	55(-18)*
Tensile Strength, psi (Chg, %)	500(-28)	621(-41)	755(-23)	866(-17)	773(-23)	674(-28)
Elongation, % (Chg, %)	240(+57)	192(-22)	132(+43)	139(+27)	114(+30)	262(+117)
Modulus @ 100%, (Chg, %)	121(-66)	287(-24)	518	503(-39)	670	325(-59)
Volume Change, %	+13.2	+10.2	+ 2.5	- 0.2	- 0.5	+14.7
Weight Change, %	+ 7.8	+5.6	+ 0.5	- 2.7	- 2.9	+ 7.0
Compression Set % of Original Deflection	51.0	45.1	67.6	82.4	74.5	64.7

AIR AND FLUID AGINGS

Fluid Age in MIL-H-83282 70 Hours @ 302°F	L449	L677	XN2046-12	XN2046-13	XN2046-14	XN2046-15
Hardness, Shore A, (chg, pts)	49(-8)	59(-4)	66(-1)	62(-1)	72(0)	60(-13)*
Tensile Strength, psi (Chg, %)	566(-18)	702(-33)	1070(+9)	1100(+6)	1030(+3)	967(+3)
Elongation, % (Chg, %)	189(+24)	220(-10)	108(+17)	120(+5)	102(+16)	194(+60)
Modulus @ 100% (Chg, %)	214(-39)	294(-23)	936	803(-1)	993	454(-42)
Volume Change, %	+ 5.0	+ 3.4	+ 2.2	+ 1.5	+ 0.1	+ 6.5
Weight Change, %	+ 3.1	+ 1.9	+ 0.1	- 0.9	- 1.8	+ 3.5
Compression Set % of Original Deflection	28.4	41.2	34.2	44.1	39.2	46.1

Fluid Age in MIL-H-83282 70 Hours @ 347°F	L449	L677	XN2046-12	XN2046-13	XN2046-14	XN2046-15
Hardness, Shore A, (Chg, pts)			62(-5)	59(-4)	67(-5)	60(-13)*
Tensile Strength, psi, (Chg, %)	Sample integrity lost		1040(+6)	1030(-1)	972(-3)	894(-5)
Elongation, % (Chg, %)			131(+42)	142(+25)	123(+40)	254(+110)
Modulus @ 100% (Chg, %)			670	569(-30)	734	356(-55)
Volume Change, %	+ 2.1*	+ 1.3	+ 2.5	+ 9.1*	+ 5.5*	+ 9.0
Weight Change, %	- 0.4	- 0.4	0	+ 2.8*	- 0.5*	+ 4.7
Compression Set % of Original Deflection	73.5	94.1	41.2	61.8	52.0	55.9

Steam Aging 240 Hours @ 302°F	L449	L677	XN2046-12	XN2046-13	XN2046-14	XN2046-15
Hardness, Shore A, (Chg, pts)	41(-16)	58(-5)	60(-7)	60(-3)	62(-10)	60(-13)*
Tensile Strength, psi (Chg, %)	155(-78)	273(-74)	394(-60)	391(-62)	328(-67)	272(-71)*
Elongation, % (Chg, %)	155(+1)	119(-51)	117(+27)	107(-6)	90(+2)	88(-27)*
Modulus @ 100% (Chg, %)	107(-70)	245(-55)	337	360	-	-
Volume Change, %	+ 6.3	+ 5.4	+52.0	+50.4	+45.8	+35.1
Weight Change, %	+ 4.7	+ 3.4	+28.6	+28.4	+24.3	+20.6

Steam Aging 240 Hours @ 347°F	L449	L677	XN2046-12	XN2046-13	XN2046-14	XN2046-15
Hardness, Shore A, (Chg, pts)			75(+8)	83(+20)	64(-8)	65(-8)*
Tensile Strength, psi (Chg, %)	Samples disintegrated					178
Elongation, % (Chg, %)						35
Modulus @ 100%, (Chg, %)						-
Volume Change, %			- 2.7	+ 5.2	+ 6.8	+11.9
Weight Change, %			- 2.9	- 3.2	+ 1.1	+ 6.1

*Variable results among specimens

HOT STRESS - STRAIN RESULTS

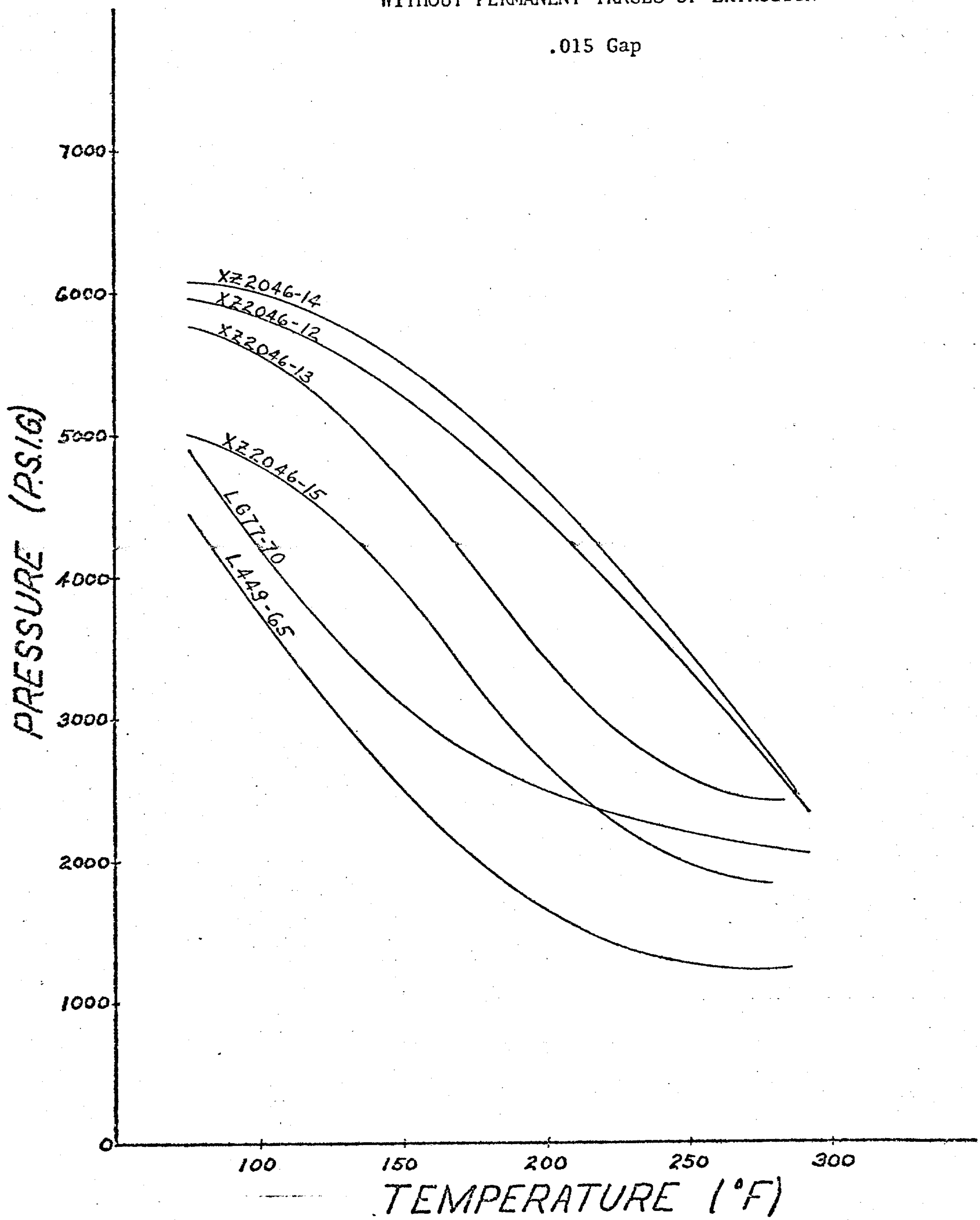
Compound	@ 275°F	@ 300°F	@ 350°F
L449-65			
Tensile	255	304	199
Elongation	82	93	68
Modulus @ 50%	138	140	131
L677-70			
Tensile	510	433	404
Elongation	150	129	119
Modulus @ 50%	162	169	167
XZ2046-12			
Tensile	593	572	565
Elongation	84	80	82
Modulus @ 50%	241	248	223
XZ2046-13			
Tensile	538	503	490
Elongation	95	90	88
Modulus @ 50%	155	159	162
XZ2046-14			
Tensile	546	538	524
Elongation	73	74	74
Modulus @ 50%	316	297	300
XZ2046-15			
Tensile	538	567	487
Elongation	127	134	130
Modulus @ 50%	138	142	133

Gregory C. Freeman
April 11, 1975

EXTRUSION RESULTS

HIGHEST PRESSURES WHICH A STATIC O-RING CAN WITHSTAND WITHOUT PERMANENT TRACES OF EXTRUSION

.015 Gap



DYNAMIC CHEW TEST RESULTS

CONDITIONS OF TEST

Sample:	2-214	Rate:	60 CPM
Rod Dia:	.998"	Temp:	275°F
Stroke:	2"	Backups:	Filled Teflon
Pressure	3000 psi		

COMPOUND	L449-65	L677-70	XZ2046-12	XZ2046-13	XZ2046-14	XZ2046-15
Fluid: MIL-H-83282						
Cycles Run	100,800	100,800	100,800	100,800	100,800	100,800
Total Leakage (cc)	11.5*	8*	16.5	18.5	15	69
Abrasion	Severe	Moderate	None	Moderate	None	Moderate
Fluid: MIL-H-5606						
Cycles	43,200	100,800	100,800	100,800	100,800	100,800
Total Leakage (cc)	Failure	6*	15	14.5	4.4	19
Abrasion	Severe	Moderate	None	None	None	Slight

*Note that an unusually low leakage combined with high abrasion signals an imminent catastrophic failure of the seal.

TABLE I

PHYSICAL PROPERTIES OF PHOSPHONITRILIC FLUOROELASTOMERS USED IN DEVELOPMENT OF O-RING COMPOUNDS

Polymer No.	K-17217[1]		K-17638[1]	
Dilute Solution Viscosity (DSV)	2.35		2.21	
% Gel	0.00		0.00	
Polymer Composition				
Mole % - OCH_2CF_3	51.3		51.3	
Mole % - $OCH_2(CF_2)_3CF_2H$	47.9		47.9	
Mole % - Cure site	0.5		1.1	
Weight % Na^+	0.05		0.07	
Weight % Cl^-	0.12		0.15	
Gum Heat Aging @ 300°F	DSV	(% Gel)	DSV	(% Gel)
24 hrs.	1.60	(0.00)	1.02	(0.00)
48	1.25	"	0.84	"
72	0.90	"	0.59	"
120	0.88	"	0.53	"
240	0.56	"	0.38	"
Specific Gravity	1.54		1.74	
ML_4/212°F	--		14	
Williams Plasticity				
1 min. (mm)	3.57		3.68	
3 min. (mm)	2.45		2.68	
Recovery (mm)	1.12		1.00	

(1) Samples were a mill blend of 9 samples (100 g.) selected at random from the entire lot.

TABLE II

PHYSICAL PROPERTIES OF O-RING STOCKS (CARBON BLACK REINFORCED-PEROXIDE CURE)(1432-2)

Compound	R190,264	R190,265	R190,266	R190,267	R190,268
PNF (K-17217)	100	100	100	100	100
FEF Black	25	25	15	20	25
MT Black	--	--	15	--	--
Austin Black	--	--	--	15	--
Stan Mag ELC	6	6	6	6	6
Epolene C-305-G	--	3	--	--	--
Stabilizer*	1	1	1	1	--
Dicup 40C	2	2	2	2	2
Mill Processing[1]	Fair	Fair-Good	Fair	Fair	Fair
Monsanto Rheometer Cure[2] **@ 335°F**					
Time to 2 pt. rise(min.)	3.1	3.7	3.0	3.7	3.6
Time to optimum cure(min.)	12.1	12.5	11.5	14.5	11.7
Minimum Torque (ip)	6.9	7.1	6.1	7.3	7.1
Maximum Torque (ip)	14.9	13.8	13.8	14.5	14.8
Cure Rate Index	11.1	11.4	11.8	9.3	12.3
@ 370°F					
Time to 2 pt. rise(min.)	1.5	1.8	1.5	1.5	1.5
Time to optimum cure(min.)	3.5	3.8	3.0	4.0	3.8
Minimum Torque (ip)	7.0	7.0	6.2	8.0	8.0
Maximum Torque (ip)	14.0	13.0	12.5	14.2	14.9
Cure Rate Index	50.0	50.0	66.7	40.0	43.5

Stress-Strain

Press Cure (min.@°F)	30/320	4/370	30/320	4/370	30/320	4/370	30/320	4/370	30/320	4/370
Post Cure (24 hr. @ 212°F)										
10% Modulus (psi)	--	42	--	42	--	35	--	38	--	42
50% Modulus (psi)	253	141	166	123	197	115	239	146	210	175
100% Modulus (psi)	1049	567	599	484	816	483	772	541	1053	959
Tensile Str. (psi)	1764	1590	1610	1530	1111	1059	1299	1310	1540	1575
E_b (%)	145	183	160	185	125	147	145	172	120	135
% Tension Set(@Break)	4	--	5	--	6	--	1	--	3	--

1. All batches stick to mill rolls, split to both rolls, fair green strength.

2. Mini-Die,(1° arc, 100 RPM)

* Bis(8-hydroxyquinolate Zinc)II -- also written as $(8\text{-}HQ)_2Zn$ elsewhere in this report.

TABLE II (CONTINUED)

PHYSICAL PROPERTIES OF O-RING STOCKS (CARBON BLACK REINFORCED-PEROXIDE CURE)(1432-2)

Compound	R190,264	R190,265	R190,266	R190,267	R190,268
Aged Stress-Strain					
Press Cure - 4' @ 370°F					
Post Cure - 24 hr. @ 212°F					
Air					
10% Modulus (psi)					
240 hr. @ 275°F	96	104	67	110	99
300°F	96	90	82	91	73
350°F	50	66	39	36	29
50% Modulus (psi)					
240 hr. @ 275°F	381	366	274	432	407
300°F	337	246	331	301	268
350°	130	120	123	99	68
100% Modulus (psi)					
240 hr. @ 275°F	1037	1094	904	939	1183
300°F	881	689	909	583	755
350°F	308	205	342	176	187
Tensile Strength (psi)					
240 hr. @ 275°F	1473	1456	1108	1261	1334
300°F	1396	1273	1036	801	1089
350°F	646	488	625	302	523
E_b (%)					
240 hr. @ 275°F	155	130	122	133	120
300°F	162	165	107	135	137
350°F	212	245	172	220	212
% Tension Set (@ Break)					
240 hr. @ 275°F	6	10	5	5	2
300°F	8	15	4	5	4
350°F	10	21	5	10	5
Hydraulic Fluid					
Mil-H-5606-B					
10% Modulus (psi)					
240 hr. @ 73°F	50	44	38	43	40
275°F	57	40	44	50	47
50% Modulus (psi)					
240 hr. @ 73°F	169	128	131	156	150
275°F	146	88	123	137	116
100% Modulus (psi)					
240 hr. @ 73°F	641	593	552	524	764
275°F	452	277	432	369	438
Tensile Strength (psi)					
240 hr. @ 73°F	1552	1388	1033	1385	1613
275°F	1311	1046	1202	1001	1161
E_b (%)					
240 hr. @ 73°F	172	160	137	180	145
275°F	207	210	167	205	182

TABLE II (CONTINUED)

PHYSICAL PROPERTIES OF O-RING STOCKS (CARBON BLACK REINFORCED-PEROXIDE CURE) (1432-2)

Compound	R190,264	R190,265	R190,266	R190,267	R190,268
Aged Shore A Hardness[3]					
Unaged	46	46	43	44	47
Air-240 hr. @ 275°F	49	50	42	47	49
Hydraulic Fluid (Mil-H-5606-B)					
240 hr. @ 73°F	45	40	41	43	46
240 hr. @ 275°F	45	35	39	41	40
% Compression Set[3,4] (ASTM D-395)					
Cylinder(70 hr. @ 275°F)	31	30	--	21	28
Plied Disks (" ")	48	46	36	39	26
Tear Strength (ppi) @ 73°F (ASTM D-639, Die B)	75	69	27	92	63
Low Temperature Properties[3]					
Gehman Torsion (ASTM-D-1053)					
Yellow Cord Wire					
Twist Angle @ 20°C	176	171	176	174	172
T_2 (°C)	-25	-44	-38	-38	-47
T_5 (°C)	-46	-50	-50	-49	-52
T_{10}(°C)	-50	-54	-53	-53	-55
T_{100} (°C)	-58	-62	-60	-60	-62
Freeze Point (°C)	-64	-66	-65	-65	-66
Youngs Modulus In Flexure[5] (ASTM D-797)					
Modulus (psi)					
@ 20°C	603	790	399	462	411
0	776	1229	490	448	666
-20	937	1301	557	673	694
-30	992	1639	622	598	766
-40	1164	2141	770	828	1281
-50	2178	3830	1078	1390	1709
-60	10230	11446	5545	8688	6944
-67	58253	48686	39539	49897	42510
YMI	-60	-59	-63	-61	-63
Recovery @ -20°C	1071	1580	1155	673	766

3. Same cure conditions as for stress-strain except press cure time doubled @ 370°F.

4. Method B, 25% Deflection

5. Also referred to as Young's Bending Modulus in other sections of this report.

TABLE II (CONTINUED)

PHYSICAL PROPERTIES OF O-RING STOCKS (CARBON BLACK REINFORCED-PEROXIDE CURE)(1432-2)

Compound	R190,264	R190,265	R190,266	R190,267	R190,268
Resistance to Hydraulic[3] Fluid (Mil-H-5606-B) (ASTM-D-471)					
Aged 240 hr. @ 73°F					
% Wt. Change	-0.16	0.97	-0.26	-0.07	-0.09
% Vol. Swell	0.20	2.47	-0.30	-0.13	0.09
% Extracted	0.42	0.01	0.40	0.31	0.32
Aged 240 hr. @ 275°F					
% Wt. Change	-1.35	4.55	-1.55	0.01	-1.21
% Vol. Swell	0.17	1.30	1.70	2.58	0.76
% Extracted	1.76	1.81	1.99	0.90	1.73
Abrasive Index[6] (ASTM D-1630)	171	181	"No Test, Too Soft"	91	158

6. Test Run on YMI Samples.

TABLE III

PHYSICAL PROPERTIES OF O-RING STOCKS (SILICA REINFORCED-PEROXIDE CURE)(1432-2)

Compound	R-190279	R-190280	R-190281	R-190282	R-19028
PNF (K-17217)	100	100	100	100	100
Silanox 101	25	25	20	20	25
Nulok 321-L	--	--	20	--	--
Mag Carb L	--	--	--	20	--
Stan Mag ELC	6	6	6	6	6
Epolene C-305-G	--	3	--	--	--
Stabilizer - $(8\text{-}HQ)_2Zn$	1	1	1	1	--
Dicup 40C	2.5	2.5	2.25	2.0	2.5
Milling Processing[1]	Fair	Fair	Fair-Good	Fair-Good	Fair
Monsanto Rheometer Cure[2]					
@ 335°F					
Time to 2 pt. rise (min.)	2.6	3.6	3.0	2.9	2.6
Time to optimum cure (min.)	15.3	16.0	12.8	21.3	13.9
Minimum Torque (ip)	7.7	8.1	9.6	9.9	8.1
Maximum Torque (ip)	21.8	18.0	22.4	25.8	20.8
Cure Rate Index	7.9	8.1	10.2	5.4	8.8
@ 370°F					
Time to 2 pt. rise (min.)	1.5	2.0	1.5	1.6	1.2
Time to optimum cure (min.)	6.6	8.6	7.5	18.3	13.4
Minimum Torque (ip)	8.7	8.3	9.7	10.1	9.5
Maximum Torque (ip)	19.8	16.1	20.6	24.0	22.5
Cure Rate Index	19.6	15.2	16.7	6.0	8.2

Stress-Strain	R-190279		R-190280		R-190281		R-190282		R-19028	
Press Cure (min. @ °F)	30/320	8/370	30/320	8/370	30/320	8/370	30/320	18/370	30/320	14/37
Post Cure (24 hr. @ 212°F)										
10% Modulus (psi)	112	68	105	67	109	65	133	94	115	[illegible]
50% Modulus (psi)	250	142	208	136	273	192	485	256	288	[illegible]
100% Modulus (psi)	808	332	596	286	862	592	1250	744	1154	[illegible]
Tensile Strength (psi)	1680	1786	1742	1567	1820	1612	1429	1362	1646	[illegible]
E_b (%)	140	205	160	210	155	170	100	140	125	[illegible]
% Tension Set (@ Break)	12	13	15	18	14	15	8	11	7	

Aged Stress-Strain	R-190279	R-190280	R-190281	R-190282	R-19028
Press Cure (min. @ 370°F)	8	8	8	18	14
Post Cure (24 hr. @ 212°F)					
Air					
10% Modulus (psi)					
240 hr. @ 275°F	123	120	133	142	110
300°F	124	117	119	143	110
350°F	116	153	130	145	113

1. All batches slightly sticky and split to both rolls, fair green strength.

2. Mini-Die, 1° arc, 100 rpm.

TABLE III (CONTINUED)

PHYSICAL PROPERTIES OF O-RING STOCKS (SILICA REINFORCED-PEROXIDE CURE)(1432-2)

Compound	R-190279	R-190280	R-190281	R-190282	R-19028
Aged Stress-Strain (continued)					
50% Modulus (psi)					
240 hr. @ 275°F	243	223	358	413	247
300°F	323	223	231	391	221
350°F	207	223	257	317	200
100% Modulus (psi)					
240 hr. @ 275°F	530	450	895	1100	556
300°F	744	442	500	945	537
350°F	370	305	441	609	378
Tensile Strength (psi)					
240 hr. @ 275°F	1606	1749	1773	1434	1667
300°F	1435	1366	1365	1353	1491
350°F	999	470	783	971	927
E_b (%)					
240 hr. @ 275°F	197	217	172	123	175
300°F	175	215	190	135	168
350°F	220	205	205	165	180
% Tension Set					
240 hr. @ 275°F	13	18	15	11	15
300°F	23	32	19	15	15
350°F	--	--	--	14	19
Hydraulic Fluid					
Mil-H-5606-B					
10% Modulus (psi)					
240 hr. @ 73°F	84	67	78	91	69
275°F	94	67	97	107	97
50% Modulus (psi)					
240 hr. @ 73°F	156	127	204	232	146
275°F	168	127	217	275	199
100% Modulus (psi)					
240 hr. @ 73°F	399	280	632	683	442
275°F	353	272	533	772	503
Tensile Strength (psi)					
240 hr. @ 73°F	1761	1519	1820	1486	1595
275°F	1626	862	1466	1292	1591
E_b (%)					
240 hr. @ 73°F	180	185	162	157	160
275°F	220	180	193	143	175
Shore A Hardness[3]					
Unaged	61	58	61	65	60
Aged in Air					
240 hr. @ 275°F	64	62	62	69	64
In Hydraulic Fluid (Mil-H-5606-B)					
240 hr. @ 73°F	57	55	53	62	56
275°F	56	44	58	64	58

3. Same cure conditions as stress-strain except press cure time @ 370°F was doubled.

TABLE III (CONTINUED)

PHYSICAL PROPERTIES OF O-RING STOCKS (SILICA REINFORCED-PEROXIDE CURE) (1432-2)

Compound	R-190279	R-190280	R-190281	R-190282	R-190283
% Compression Set[3,4] (ASTM D-395)					
Cylinder (70 hr. @ 275°F)	60	59	69	54	54
Plied (70 hr. @ 275°F)	62	67	76	64	62
Tear Strength (ppi) @ 73°F[3] (ASTM D-639, Die B)	76	86	93	95	68
Low Temp. Properties[3] Gehman Torsion (ASTM D-1053) Yellow Cord Wire					
Twist Angle @ 20°C	163	168	168	163	162
T_2 (°C)	-35	-31	-33	-36	-39
T_5 (°C)	-43	-44	-42	-43	-46
T_{10} (°C)	-48	-49	-46	-47	-50
T_{100} (°C)	-62	-62	-58	-58	-61
Freeze Point (°C)	-65	-65	-64	-62	-62
Youngs Modulus in Flexure (ASTM D-797) Modulus (psi)					
@ 20°C	1335	1008	1143	1147	681
0°C	1880	1680	1559	1622	1362
-20°C	2496	2232	2129	2215	1702
-30°C	3324	2778	2744	2855	2189
-40°C	4432	3820	4042	4282	3192
-50°C	7388	6721	6431	7695	5532
-60°C	16855	16371	16769	19629	14982
-67°C	91021	91681	90556	67300	44947
YMI°C	-55	-56	-56	-54	-57
Recovery @ -20°C	2784	2500	2286	3059	1964
Resistance to Hydraulic Fluid[3] Mil-H-5606-B) ASTM-D-471)					
Aged 240 hr. @ 73°F					
% Wt. Change	0.76	0.43	0.00	0.00	0.00
% Volume Swell	1.69	1.49	0.34	0.21	0.15
% Extracted	--	--	--	--	--
Aged 240 hr. @ 275°F					
% Wt. Change	-0.52	9.97	-0.08	-0.20	-0.31
% Volume Swell	2.64	26.15	2.48	1.93	2.53
% Extracted	1.42	-6.49	0.82	0.92	1.08
Abrasive Index[5] (ASTM D-1630)	75	146	61	32	96

4. Method B, 25% Deflection.

5. Test run on YMI samples.

TABLE IV

COMPOUNDING STUDIES TO IMPROVE MILL PROCESSING AND HARDNESS OF O-RING STOCKS

Compound R-191-	920	921	922	923	924
K-17217	100	100	100	100	100
FEF Black	25	25	25	25	--
Graphite (Superior 5033X)	--	--	15	--	--
Quso WR 82	--	--	--	--	25
Mistron Vapor	--	--	--	15	--
AC Polyethylene	--	5	--	--	--
Stabilizer - $(8\text{-HQ})_2Zn$	1	1	1	1	1
Chem Link 30	3	3	--	--	--
Stan Mag ELC	6	6	6	6	6
Dicup 40C	3	3	3	3	3
Mill Processing(1)	Fair	Fair-Good	Fair	Fair	Fair
Monsanto Rheometer Cure(2)					
@ 335°F					
Time to 2 pt. Rise (min.)	1.5	1.5	2.5	3.0	1.9
Time to Optimum Cure (min.)	13.3	15.8	11.8	10.8	23.8
Minimum Torque (ip)	6.4	5.9	8.5	8.6	7.5
Maximum Torque (ip)	21.0	19.7	22.0	21.0	27.2
Cure Rate Index	8.4	7.0	7.0	12.8	4.6
@ 370°F					
Time to 2 pt. Rise (min.)	0.8	0.8	1.0	1.2	0.8
Time to Optimum Cure (min.)	5.0	4.3	4.5	3.5	14.6
Minimum Torque (ip)	7.0	8.5	10.5	13.8	10.0
Maximum Torque (ip)	20.9	21.8	25.8	24.0	30.8
Cure Rate Index	23.8	28.6	28.6	43.5	7.2
Stress-Strain					
(Press Cure - 30 min. @ 320°F) (Post Cure - 24 hr. @ 212°F)					
10% M (psi)	43	144	166	86	97
50% M (psi)	289	546	921	368	688
100% M (psi)	--	1505	--	996	--
Tensile Strength (psi)	684	1549	1489	1601	1161
E_b (%)	86	108	80	167	70
% Tension Set (@ Break)	2	10	6	15	4

(1) All batches are slightly sticky and split to both rolls; fair green strength.

(2) Mini Die, 1° Arc, 100 RPM.

TABLE IV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE MILL PROCESSING AND HARDNESS OF O-RING STOCKS

Compound R-191-	920	921	922	923	924
Aged Stress-Strain					
Press Cure (min. @ 370°F) Post Cure (24 hr. @ 212°F)	5	5	5	4	15
10% M (psi) (Unaged)	145	152	221	189	195
240 hr. @ 275°F (Air)	93	116	110	184	170
" " @ 300°F "	168	162	211	179	208
672 hr. @ " " "	135	139	213	159	141
1000 hr. @ " " "	91	120	132	124	123
240 hr. @ 350°F "	131	151	191	187	190
672 hr. @ " " "	71	177	91	86	182
1000 hr. @ " " "	36	119	75	67	236
240 hr. @ 73°F (HF)(3)	93	107	157	125	63
" " @ 275°F "	103	113	149	121	59
50% M (psi) (Unaged)	655	461	624	561	935
240 hr. @ 275°F (Air)	423	333	495	580	806
" " @ 300°F "	669	439	594	554	1034
672 hr. @ " " "	566	320	534	461	668
1000 hr. @ " " "	326	245	413	361	617
240 hr. @ 350°F "	377	283	423	404	881
672 hr. @ " " "	154	--	194	158	324
1000 hr. @ " " "	111	--	152	98	343
240 hr. @ 73°F (HF)	508	381	555	487	399
" " @ 275°F "	446	387	476	422	336
100% M (psi) (Unaged)	--	1151	1118	1047	--
240 hr. @ 275°F (Air)	1388	891	908	979	--
" " @ 300°F "	--	1014	980	914	--
672 hr. @ " " "	--	655	--	717	--
1000 hr. @ " " "	626	482	661	602	1097
240 hr. @ 350°F "	815	505	647	589	--
672 hr. @ " " "	271	--	267	205	--
1000 hr. @ " " "	184	--	191	113	--
240 hr. @ 73°F (HF)	--	1054	1082	1018	--
" " @ 275°F "	115	1018	866	801	1056

(3) Hydraulic Fluid Mil H-5606-C.

TABLE IV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE MILL PROCESSING AND HARDNESS OF O-RING STOCKS

Compound R-191-	920	921	922	923	924
Aged Stress-Strain (contd.)					
Tensile Strength (psi) (Unaged)	1454	1244	1140	1204	1065
240 hr. @ 275°F (Air)	1413	1212	1089	1074	1124
" " @ 300°F "	1303	1178	994	1023	1216
672 hr. @ " " "	1295	871	803	761	1100'
1000 hr. @ " " "	626	855	768	684	1097
240 hr. @ 350°F "	864	665	671	611	1089
672 hr. @ " " "	327	189	311	221	431
1000 hr. @ " " "	236	124	200	120	368
240 hr. @ 73°F (HF)	1377	1124	1155	1313	1120
" " @ 275°F "	1317	1174	1043	1127	1029
E_b (%) (Unaged)	80	112	110	130	60
240 hr. @ 275°F (Air)	100	135	130	125	70
" " @ 300°F "	90	120	107	137	62
672 hr. @ " " "	100	140	90	115	85
1000 hr. @ " " "	125	170	150	135	100
240 hr. @ 350°F "	110	147	113	132	67
672 hr. @ " " "	145	40	145	130	85
1000 hr. @ " " "	170	37	130	150	72
240 hr. @ 73°F (HF)	97	110	112	145	87
" " @ 275°F "	112	117	133	180	100
% Tension Set (@ Break)(Unaged)	3	7	10	22	3
240 hr. @ 275°F (Air)	6	15	15	17	5
" " @ 300°F "	3	13	11	19	5
672 hr. @ " " "	3	12	11	19	7
1000 hr. @ " " "	4	20	14	21	7
240 hr. @ 350°F "	4	21	14	19	6
672 hr. @ " " "	20	18	27	53	37
1000 hr. @ " " "	32	16	25	53	37
240 hr. @ 73°F (HF)	2	5	9	21	5
" " @ 275°F "	2	3	1	20	6
Shore A Hardness (Unaged)(4)	61	67	63	63	61
240 hr. @ 275°F (Air)	62	70	70	68	66
" " @ 300°F "	65	70	70	70	67
" " @ 350°F "	64	68	65	67	65
" " @ 73°F (HF)(3)	61	65	62	62	63
" " @ 275°F "	60	56	63	64	62

(4) R-191,920, 921 and 922: Press Cure - 10' @ 370°F, Post Cure - 24 hr. @ 212°F
R-191,923: Press Cure - 8' @ 370°F, Post Cure - 24 hr. @ 212°F
R-191,924: Press Cure -30' @ 370°F, Post Cure - 24 hr. @ 212°F

TABLE IV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE MILL PROCESSING AND HARDNESS OF O-RING STOCKS

Compound R-191-	920	921	922	923	924
% Compression Set (4,5) (70 hr. @ 275°F)					
Cylinders	13	11	19	35	15
Plied Disks	34	15	22	45	31
Tear Strength (ppi) (4) (ASTM D-369, Die B)	60	80	68	104	32
Abrasive Index (4)(6) (ASTM D-1630)	58	63	43	28	42
Low Temperature Properties (4)					
Gehman Torsion (ASTM D-1053)					
Black Torsion Wire					
Isooctane Coolant					
Twist Angle @ 73°F (Deg.)	154	140	154	148	153
T_2 (°C)	-39	-23	-38	-28	-34
T_5 (°C)	-46	-44	-47	-46	-46
T_{10} (°C)	-50	-51	-52	-51	-53
T_{100} (°C)	-61	-64	-62	-62	-63
Freeze Point (°C)	-60	-64	-62	-62	-63
Youngs' Modulus in Flexure (ASTM D-797)					
Modulus (psi) @:					
20°C	878	1938	1533	1397	933
0°C	1120	2763	1703	1787	1015
-20°C	1312	3807	1796	2148	1190
-30°C	1397	4619	1982	2606	1278
-40°C	1934	6822	2653	3591	1917
-50°C	2888	8528	5366	5586	3486
-60°C	9582	16174	12776	15858	11505
-67°C	59575	58633	67459	98320	92044
YMI °C	-61	-54	-58	-57	-60
Recovery	1312	4441	1854	2539	1190

(5) ASTM D-395, Method B, 25% Deflection, Disks Cut From 6" x 6" x 0.075" Slab.

(6) Test Run on YMI Specimens.

TABLE IV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE MILL PROCESSING AND HARDNESS OF O-RING STOCKS

Compound R-191-	920	921	922	923	924
Resistance to Hydraulic Fluid (ASTM D-471) (Mil-H-5606-C)					
240 hr. @ 73°F					
% Wt. Change	-0.16	0.36	-0.11	0.13	-0.10
% Vol. Swell	0.26	1.19	0.32	0.67	0.67
% Extracted	0.30	0.23	0.36	0.36	0.30
240 hr. @ 275°F					
% Wt. Change	-0.70	2.90	-0.36	-0.61	-0.35
% Vol. Swell	0.83	12.74	2.18	1.31	1.73
% Extracted	1.17	1.72	0.74	0.85	0.66

TABLE V

O-RING STOCKS SUBMITTED TO PARKER SEAL FOR EVALUATION

Compound R-191-	941	959
K-17217	100	100
FEF Black	20	--
Austin Black	20	--
Quso WR 82	--	25
Stan Mag ELC	6	6
Stabilizer - $(8\text{-HQ})_2Zn$	1	1
Dicup 40C	2.5	2.0
Mill Processing(1)	Fair	Fair
Monsanto Rheometer Cure(2)		
@ 335°F		
Time to 2 pt. Rise (min.)	3.0	2.0
Time to Optimum Cure (min.)	10.8	12.3
Minimum Torque (ip)	8.2	7.8
Maximum Torque (ip)	16.8	21.0
Cure Rate Index	12.8	9.7
@ 370°F		
Time to 2 pt. Rise (min.)	1.5	1.3
Time to Optimum Cure (min.)	4.0	8.3
Minimum Torque (ip)	9.2	8.0
Maximum Torque (ip)	17.0	21.3
Cure Rate Index	40.0	14.2
Stress-Strain		
Press Cure (6 min. @ 370°F)		
Post Cure (24 hr. @ 212°F)		
10% M (psi)	109	115
50% M (psi)	484	550
100% M (psi)	1120	--
Tensile Strength (psi)	1191	1272
E_b (%)	110	90
% Tension Set (@ Break)	3	3

(1) Both stocks stick to mill rolls and split to both rolls, fair green strength.

(2) Mini-Die, 1° Arc, 100 RPM.

TABLE V (CONTINUED)

O-RING STOCKS SUBMITTED TO PARKER SEAL FOR EVALUATION

Compound R-191-	941		959	
Aged Stress-Strain				
Press Cure (min. @ 370°F)	6		10	
Post Cure (24 hr. @ 212°F)				
10% M (psi) (Unaged)	--		--	
240 hr. @ 275°F (Air)	162		135	
" " @ 300°F "	125		127	
" " @ 350°F "	60		108	
Hydraulic Fluid Mil-H-	5606-C	83282	5606-C	83282
240 hr. @ 275°F	77	57	66	83
480 hr. @ " "	79	64	84	65
672 hr. @ " "	66	75	76	64
1000 hr. @ " "	53	55	58	57
50% M (psi) (Unaged)	423		400	
240 hr. @ 275°F (Air)	668		643	
" " @ 300°F "	440		623	
" " @ 350°F "	134		396	
Hydraulic Fluid				
240 hr. @ 275°F	376	298	353	482
480 hr. @ " "	302	272	423	306
672 hr. @ " "	217	282	350	292
1000 hr. @ " "	178	183	242	230
100% M (psi) (Unaged)	910		1295	
240 hr. @ 275°F (Air)	--		--	
" " @ 300°F "	818		--	
" " @ 350°F "	215		882	
Hydraulic Fluid				
240 hr. @ 275°F	897	712	1091	--
480 hr. @ " "	633	607	1205	1011
672 hr. @ " "	420	590	984	865
1000 hr. @ " "	367	393	737	710

TABLE V (CONTINUED)

O-RING STOCKS SUBMITTED TO PARKER SEAL FOR EVALUATION

Compound R-191-	941		959	
Aged Stress-Strain (contd.)				
Tensile Strength (psi)(Unaged)	1233		1382	
240 hr. @ 275°F (Air)	1106		1062	
" " @ 300°F "	868		876	
" " @ 350°F "	300		939	
Hydraulic Fluids Mil-H-	5606-C	83282	5606-C	83282
240 hr. @ 275°F	1033	1078	1252	1029
480 hr. @ " "	784	782	1317	1169
672 hr. @ " "	613	708	1189	1166
1000 hr. @ " "	586	500	1159	1019
E_b (%) (Unaged)	113		105	
240 hr. @ 275°F (Air)	87		87	
" " @ 300°F "	110		72	
" " @ 350°F "	167		115	
Hydraulic Fluid				
240 hr. @ 275°F	120	147	115	87
480 hr. @ " "	130	137	112	110
672 hr. @ " "	155	125	120	127
1000 hr. @ " "	165	127	145	135
% Tension Set (@ Break)(Unaged)	3		4	
240 hr. @ 275°F (Air)	4		6	
" " @ 300°F "	2		6	
" " @ 350°F "	19		11	
Hydraulic Fluid				
240 hr. @ 275°F	2	4	4	2
480 hr. @ " "	3	4	3	2
672 hr. @ " "	7	3	4	3
1000 hr. @ " "	7	5	7	8
Shore A Hardness (3)(4)				
Unaged	50		55	
240 hr. @ 275°F (Air)	50		52	
" " @ 300°F "	47		52	
" " @ 350°F "	35		54	
Hydraulic Fluid				
240 hr. @ 275°F	41	40	42	40
480 hr. @ " "	40	40	41	40
672 hr. @ " "	40	41	42	42
1000 hr. @ " "	40	41	45	42

(3) Press Cures 16' @ 370°F for R-191,959: 8' @ 370°F for R-191,959; both post-cured 240 hr. @ 212°F.

(4) Measured on Stacked Tensile Strips

TABLE V (CONTINUED)

O-RING STOCKS SUBMITTED TO PARKER SEAL FOR EVALUATION

Compound R-191-	941	959
Compression Set[3]		
(ASTM D-395, Method B)		
% Set (70 hr. @ 275°F)	19	25
% Set (70 hr. @ 300°F)	30	28
Tear Strength (ppi)[3]		
(ASTM D-639, Die B)		
@ 275°F	22	10
Hot Stress-Strain (@ 275°F)[3]		
100% M (psi)	570	580
Tensile Strength (psi)	735	666
E_b (%)	115	113
Abrasive Index[5]	49	55
(ASTM D-1630)		
Low Temperature Properties		
Gehman Torsion[3]		
(ASTM D-1053)		
Black Torsion Wire		
Isooctane Coolant		
Twist Angle @ 20°C	164	155
T_2 (°C)	-41	-42
T_5 (°C)	-46	-48
T_{10} (°C)	-50	-52
T_{100} (°C)	-60	-63
Freeze Point (°C)	-61	-61
Youngs' Modulus in Flexure[3]		
(ASTM D-797)		
Modulus (psi) A:		
20°C	744	842
0°C	843	811
-20°C	930	1052
-30°C	1044	974
-40°C	1542	1175
-50°C	2290	1959
-60°C	11995	10521
-67°C	67858	71427
YMI	-60	-60
Recovery (psi)	1038	1052

(5) Test Run on YMI Samples.

TABLE V (CONTINUED)

O-RING STOCKS SUBMITTED TO PARKER SEAL FOR EVALUATION

Compound R-191-	941		959	
Resistance to Hydraulic Fluids (ASTM D-471)(3) Mil-H-	5606-C	83282	5606-C	83282
240 hr. @ 275°F				
% Wt. Change	-0.21	-1.41	-0.74	-1.25
% Vol. Swell	2.18	1.22	1.52	1.02
% Extracted	0.63	0.87	1.67	1.01
480 hr. @ 275°F				
% Wt. Change	0.00	-0.75	-0.17	-0.93
% Vol. Swell	1.63	0.31	2.08	1.04
% Extracted	0.28	0.75	0.44	0.86
672 hr. @ 275°F				
% Wt. Change	0.19	-0.49	0.09	-0.71
% Vol. Swell	1.84	0.76	2.92	1.33
% Extracted	-0.12	0.29	0.26	0.39
1000 hr. @ 275°F				
% Wt. Change	0.79	0.26	0.34	0.20
% Vol. Swell	2.35	1.93	3.10	3.15
% Extracted	-0.70	-0.46	-0.24	-0.45

TABLE VI

QUSO WR 82 REINFORCED O-RING COMPOUNDS

Compound R-191-	972	973	974	975	976
K-17217	100	100	100	100	100
Quso WR 82	25	30	35	40	30
Stan Mag ELC	6	6	6	6	6
Stabilizer- $(8\text{-HQ})_2Zn$	1	1	1	1	2
Dicup 40C	1	1	1	1	1
Mill Processing[(1)]	Fair	Fair	Fair-Good	Fair-Good	Fair
Monsanto Rheometer Cure[(2)]					
@ 335°F					
Time to 2 pt. Rise (min.)	3.0	2.7	2.4	2.7	2.8
Time to Optimum Cure (min.)	18.3	19.8	16.3	32.3	29.3
Minimum Torque (ip)	7.3	7.5	8.6	9.3	7.8
Maximum Torque (ip)	16.0	21.2	29.0	27.8	21.0
Cure Rate Index	6.5	5.9	7.2	3.4	3.8
@ 370°F					
Time to 2 pt. Rise (min.)	1.3	1.4	1.2	1.0	1.3
Time to Optimum Cure (min.)	8.6	13.3	12.8	20.3	9.8
Minimum Torque (ip)	6.9	8.8	11.2	11.0	9.5
Maximum Torque (ip)	15.8	22.0	26.9	31.0	22.0
Cure Rate Index	13.7	8.4	8.6	5.1	11.7
Stress-Strain					
Press Cure - 30' @ 320°F					
Post Cure - 24 hr. @ 212°F					
50% M (psi)	305	585	545	575	440
100% M (psi)	875	1340	945		1280
Tensile Strength (psi)	1275	1465	1070	770	1460
E_b (%)	130	110	120	95	105
% Tension Set (@ Break)	7	10	13	13	8

(1) All batches are slightly sticky and split to both rolls; fair green strength.

(2) Mini-Die, 1° Arc, 100 RPM.

TABLE VI (CONTINUED)

QUSO WR 82 REINFORCED O-RING COMPOUNDS

Compound R-191-	972	973	974	975	976
Tensile Strength (psi)					
(Unaged)	1427	1389	1008	824	1403
240 hr. @ 275°F (Air)	1386	1303	1055	797	1503
672 hr. @ 275°F (Air)	1300	1336	993	857	1495
240 hr. @ 300°F (Air)	1383	1242	977	862	1380
672 hr. @ 300°F (Air)	-	-	-	708	-
240 hr. @ 350°F (Air)	1063	921	720	558	1138
240 hr. @ 275°F (HF)	1502	1270	975	708	1408
672 hr. @ 275°F (HF)	1313	1183	863	666	1346
E_b (%) (Unaged)	168	160	162	187	125
240 hr. @ 275°F (Air)	157	172	170	143	175
672 hr. @ 275°F (Air)	162	182	150	173	160
240 hr. @ 300°F (Air)	170	177	175	157	165
672 hr. @ 300°F (Air)	-	-	-	117	-
240 hr. @ 350°F (Air)	190	185	147	105	165
240 hr. @ 275°F (HF)	186	177	177	152	177
672 hr. @ 275°F (HF)	165	172	182	165	170
% Tension Set (@ Break)					
(Unaged)	5	8	14	26	5
240 hr. @ 275°F (Air)	10	12	19	21	16
672 hr. @ 275°F (Air)	9	13	17	31	11
240 hr. @ 300°F (Air)	10	14	19	26	12
672 hr. @ 300°F (Air)	-	-	-	23	-
240 hr. @ 350°F (Air)	13	21	27	28	18
240 hr. @ 275°F (HF)	3	14	6	19	8
672 hr. @ 275°F (HF)	7	9	20	24	9
Shore A Hardness (Unaged)(4)	47	55	62	72	54
240 hr. @ 275°F (Air)	51	61	69	75	59
672 hr. @ 275°F (Air)	52	59	70	78	58
240 hr. @ 300°F (Air)	53	63	70	80	58
672 hr. @ 300°F (Air)	-	-	-	77	-
240 hr. @ 350°F (Air)	52	61	71	77	59
240 hr. @ 275°F (HF)	47	55	58	71	52
480 hr. @ 275°F (HF)	-	-	-	-	52
672 hr. @ 275°F (HF)	46	54	62	69	53

(4) Press cures @ 370°F; R-191,972 (18 min.), 973 (26 min.), 974 (26 min.), 975 (40 min.) and 976 (20 min.). All post cured 24 hr. @ 212°F.

TABLE VI(CONTINUED)

QUSO WR 82 REINFORCED O-RING COMPOUNDS

Compound R-191-	972	973	974	975	976
Aged Stress-Strain					
Press Cure (min. @ 370°F)	9	13	13	20	10
Post Cure (24 hr. @ 212°F)					
10% M (psi)(Unaged)	31	47	80	129	70
240 hr. @ 275°F (Air)	41	60	106	144	63
672 hr. @ 275°F (Air)	44	58	102	185	54
240 hr. @ 300°F (Air)	45	52	100	159	64
672 hr. @ 300°F (Air)	-	-	-	177	-
240 hr. @ 350°F (Air)	52	64	110	195	60
240 hr. @ 275°F (HF)(3)	29	36	64	103	37
672 hr. @ 275°F (HF)	60	68	107	156	71
50% M (psi)(Unaged)	100	208	295	378	314
240 hr. @ 275°F (Air)	153	246	386	457	212
672 hr. @ 275°F (Air)	144	231	382	510	202
240 hr. @ 300°F (Air)	147	212	363	496	226
672 hr. @ 300°F (Air)	-	-	-	501	-
240 hr. @ 350°F (Air)	154	230	341	447	196
240 hr. @ 275°F (HF)	72	134	247	301	123
672 hr. @ 275°F (HF)	172	218	295	354	215
100% M (psi) (Unaged)	439	835	748	658	1184
240 hr. @ 275°F (Air)	649	803	797	715	742
672 hr. @ 275°F (Air)	574	763	795	720	766
240 hr. @ 300°F (Air)	575	730	748	740	825
672 hr. @ 300°F (Air)	-	-	-	671	-
240 hr. @ 350°F (Air)	465	588	621	558	569
240 hr. @ 275°F (HF)	343	597	772	630	514
672 hr. @ 275°F (HF)	553	649	629	601	658

(3) Mil-H-5606-C

TABLE VI (CONTINUED)

QUSO WR 82 REINFORCED O-RING COMPOUNDS

Compound R-191-	972	973	974	975	976
Compression Set[4] (70 hr. @ 275°F) (ASTM D-395, Method B)					
% Set (Cylinder)	20	26	35	42	25
% Set (Plied Disk)[5]	34	34	54	66	38
Tear Strength (ppi)[4] (ASTM D-369, Die B)	58	101	118	121	87
Abrasive Index[4][6] (ASTM D-1630)	--	64	51	42	86
Low Temperature Properties					
Youngs' Modulus in Flexure[4] (ASTM D-797)					
Modulus (psi) @:					
20°C	--	1777	1918	1948	960
0°C	--	1110	1918	2191	1016
-20°C	--	1225	2397	2821	1191
-30°C	--	1480	2663	3224	1280
-40°C	--	1754	3425	4286	1707
-50°C	--	2961	5413	6575	3142
-60°C	--	8460	13319	15585	9292
-67°C	--	80178	87911	93513	77998
YMI	--	-61	-58	-57	-61
Recovery	--	1225	2369	2821	1191
Gehman Torsion[4]					
Black Torsion Wire					
Isooctane Coolant					
Twist Angle @ 20°C (Deg.)	161	159	142	129	158
T_2 (°C)	-43	-39	-36	-18	-31
T_5 (°C)	-48	-45	-45	-39	-45
T_{10} (°C)	-51	-49	-49	-46	-49
T_{100} (°C)	-60	-59	-61	-61	-59
Freeze Point (°C)	-60	-59	-58	-58	-59
Resistance to Hydraulic Fluid (4) (ASTM D-471)					
240 hr. @ 275°F in Mil-H-5606-C					
% Wt. Change	0.16	0.19	0.10	0.18	0.02
% Vol. Swell	1.86	2.40	2.58	2.86	2.56
% Extracted	0.22	0.24	0.06	0.11	0.02

(5) Disks cut from 6" x 6" x 0.075" Slabs

(6) Test Run on YMI Sample

TABLE VII

SILICA REINFORCED O-RING COMPOUNDS - THE EFFECTS OF PEROXIDE AND QUSO WR 82--SILANOX 101 LEVELS ON PHYSICAL PROPERTIES

Compound R-191-	977	978	979	980	981
K-17217	100	100	100	100	100
Quso WR 82	30	30	30	20	25
Silanox 101	--	--	--	10	--
Cab-O-Sil S-17	--	--	--	--	5
Stan Mag ELC	6	6	--	6	6
Tribase	--	--	6	--	--
Stabilizer - $(8\text{-HQ})_2Zn$	1.0	1.0	1.0	1.0	1.0
Dicup 40C	0.5	1.5	1.0	1.0	1.0
Mill Processing(1)	Fair	Fair	Fair	Fair	Fair
Monsanto Rheometer Cure(2)					
@ 335°F					
Time to 2 pt. Rise (min.)	3.0	1.8	1.5	2.0	1.5
Time to Optimum Cure (min.)	16.3	17.3	16.5	13.3	63.8
Minimum Torque (ip)	7.5	7.0	6.0	7.6	13.8
Maximum Torque (ip)	16.0	22.6	18.0	17.0	41.0
Cure Rate Index	7.5	6.4	6.6	8.8	1.6
@ 370°F					
Time to 2 pt. Rise (min.)	2.0	1.1	1.5	1.3	1.0
Time to Optimum Cure (min.)	14.3	21.3	6.5	3.5	29.8
Minimum Torque (ip)	7.2	7.4	7.0	7.4	15.0
Maximum Torque (ip)	14.8	25.0	18.0	15.2	38.0
Cure Rate Index	8.1	4.9	20.0	83.3	3.5
Stress-Strain					
Press Cure (30' @ 320°F)					
Post Cure - 24 hr. @ 212°F					
10% M (psi)	94	149	100	113	204
50% M (psi)	388	870	341	363	643
100% M (psi)	1120	--	1107	1477	1460
Tensile Strength (psi)	1586	1557	1586	1874	1634
E_o (%)	160	75	135	125	120
% Tension Set (@ Break)	7	3	6	2	12

(1) All batches are slightly sticky and split to both rolls; fair green strength.

(2) Mini-Die, 1° Arc, 100 RPM.

TABLE VII (CONTINUED)

SILICA REINFORCED O-RING COMPOUNDS - THE EFFECTS OF PEROXIDE AND QUSO WR 82--SILANOX 101 LEVELS ON PHYSICAL PROPERTIES

Compound R-191-	977	978	979	980	981
Aged Stress-Strain					
Press Cure (min. @ 370°F)	10	21	7	4	60
Post Cure - 24 hr. @ 212°F					
10% M (psi)(Unaged)	52	81	72	63	112
240 hr. @ 275°F (Air)	82	69	64	61	164
672 hr. @ " " "	59	77	83	92	167
240 hr. @ 300°F "	76	69	156	105	146
" " @ 350°F "	84	120	51	136	147
240 hr. @ 275°F (HF)(3)	38	46	46	39	81
672 hr. @ " " "	62	85	73	73	131
50% M (psi)(Unaged)	150	439	260	210	374
240 hr. @ 275°F (Air)	218	283	219	173	475
672 hr. @ " " "	159	329	306	265	482
240 hr. @ 300°F "	211	299	524	312	340
" " @ 350°F "	196	402	59	261	275
240 hr. @ 275°F (HF)	85	220	141	100	239
672 hr. @ " " "	126	299	161	163	344
100% M (psi)(Unaged)	583	1480	841	852	1103
240 hr. @ 275°F (Air)	682	1065	613	552	1182
672 hr. @ " " "	468	1137	718	774	1095
240 hr. @ 300°F "	632	1095	1047	975	899
" " @ 350°F "	431	935	69	483	517
240 hr. @ 275°F (HF)	297	1014	415	419	744
672 hr. @ " " "	342	961	382	438	863
Tensile Strength (psi) (Unaged)	1396	1523	1409	1551	1399
240 hr. @ 275°F (Air)	1575	1593	1412	1859	1511
672 hr. @ " " "	1333	1458	1187	1597	1416
240 hr. @ 300°F "	1375	1516	1354	1665	1480
" " @ 350°F "	873	1093	100	819	854
240 hr. @ 275°F (HF)	1475	1499	1386	1663	1465
672 hr. @ " " "	1233	1375	1202	1463	1267
E_b (%) (Unaged)	103	105	150	140	132
240 hr. @ 275°F (Air)	207	140	200	187	137
672 hr. @ " " "	237	125	185	165	142
240 hr. @ 300°F "	210	130	157	147	167
" " @ 350°F "	210	122	292	180	180
240 hr. @ 275°F (HF)	250	130	217	190	167
672 hr. @ " " "	235	137	232	190	150

(3) Mil-H-5606-C

TABLE VII (CONTINUED)

SILICA REINFORCED O-RING COMPOUNDS - THE EFFECTS OF PEROXIDE AND QUSO WR 82--SILANOX 101 LEVELS ON PHYSICAL PROPERTIES

Compound R-191-	977	978	979	980	981
% Tension Set (@ Break)					
(Unaged)	11	4	10	4	10
240 hr. @ 275°F (Air)	15	9	18	13	12
672 hr. @ " " "	20	11	20	11	12
240 hr. @ 300°F "	18	11	17	11	17
" " @ 350°F "	26	17	200	52	31
240 hr. @ 275°F (HF)	13	7	15	7	10
672 hr. @ " " "	14	6	24	10	9
Shore A Hardness (Unaged)(4)	45	65	49	49	66
240 hr. @ 275°F (Air)	51	61	57	55	71
672 hr. @ " " "	54	62	65	59	78
240 hr. @ 300°F "	51	62	58	57	71
" " @ 350°F "	54	62	61	58	67
240 hr. @ 275°F (HF)	45	56	51	49	65
672 hr. @ " " "	44	55	52	48	64
Compression Set(4)					
(ASTM D-395, Method B)					
70 hr. @ 275°F					
% Set (Cylinder)	28	18	62	29	32
% Set (Plied Disk)(5)	42	34	72	44	47
Tear Strength (ppi)	139	77	107	77	92
ASTM D-369 (Die B)					
Abrasive Index	--(6)	88	--(6)	--(6)	39
(ASTM D-1630)					
Low Temperature Properties					
Gehman Torsion(4)					
(ASTM D-1053)					
Black Torsion Wire					
Isooctane Coolant					
Twist Angle @ 20°C (Deg.)	159	155	144	158	163
T_2 (°C)	-35	-35	-33	-36	-38
T_5 (°C)	-44	-45	-43	-44	-45
T_{10} (°C)	-49	-50	-48	-47	-49
T_{100} (°C)	-60	-61	-61	-57	-59
Freeze Point (°C)	-59	-59	-59	-58	-60

(4) Press Cure (min. @ 370°F) - 977 (28), 978 (42), 979 (14), 980 (8), 981 (60). All stocks post cured 24 hr. @ 212°F.

(5) Cut from 6" x 6" x 0.075" slabs

(6) Too soft to test

TABLE VII(CONTINUED)

SILICA REINFORCED O-RING COMPOUNDS -- THE EFFECTS OF PEROXIDE AND QUSO WR 82--SILANOX 101 LEVELS ON PHYSICAL PROPERTIES

Compound R-191-	977	978	979	980	981
Youngs' Modulus in Flexure (4)					
(ASTM D-797)					
Modulus (psi) @:					
20 (°C)	774	833	828	748	1551
0 (°C)	841	969	966	775	1939
-20 (°C)	887	1081	1073	844	2101
-30 (°C)	1064	1163	1122	1083	2621
-40 (°C)	1282	1499	1325	1551	3217
-50 (°C)	2524	2812	3198	2618	5791
-60 (°C)	7328	8999	9350	8056	13573
-67 (°C)	58798	61873	85024	57604	106171
YMI (°C)	-62	-61	-61	-62	-58
Recovery (psi)	916	1081	1073	1007	2246
Resistance to Hydraulic Fluid (4)					
(ASTM D-471)					
240 hr. @ 275°F in Mil-H-5606-C					
% Wt. Change	0.18	-0.19	-0.21	0.02	0.24
% Vol. Swell	2.64	1.90	2.12	2.55	2.92
% Extracted	0.06	0.27	0.28	0.11	0.12

TABLE VIII

MILL PROCESSING AND POST CURE STUDIES ON O-RING STOCKS

Compound R-193-	222	223	213	214
K-17217	100	100	100	100
Quso WR 82	20	20	--	--
Silanox 101	10	10	--	--
FEF Black	--	--	30	30
Stan Mag ELC	6	6	6	6
Stabilizer - $(8\text{-HQ})_2Zn$	1	1	1	1
Silastic 410	--	6	--	6
Dicup 40C	1.1	1.1	2	2
Mill Processing[(1)]	Fair	Fair-Good	Fair	Excellent
Monsanto Rheometer Cure[(2)]				
@ 335°F				
Time to 2 pt. Rise (min.)	2.1	2.3	2.4	2.6
Time to Optimum Cure (min.)	10.3	12.8	11.6	12.3
Minimum Torque (ip)	8.7	8.6	10.2	10.0
Maximum Torque (ip)	18.4	19.3	20.8	21.4
Cure Rate Index	12.2	9.5	10.9	10.3
@ 370°F				
Time to 2 pt. Rise (min.)	1.3	1.4	1.3	1.1
Time to Optimum Cure (min.)	3.6	4.3	3.5	3.8
Minimum Torque (ip)	9.0	8.0	9.0	10.0
Maximum Torque (ip)	17.5	17.0	18.5	20.5
Cure Rate Index	43.5	34.5	45.4	37.0
Stress-Strain				
Press Cure - 30' @ 320°F				
Post Cure - 24 hr. @ 212°F				
10% M (psi)	46	50	62	71
50% M (psi)	130	156	318	376
100% M (psi)	432	518	1385	1317
Tensile Strength (psi)	1555	1693	1718	1650
E_b (%)	180	180	130	130
% Tension Set (@ Break)	6	9	5	6

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(1) R-193- 222, sticky and splits to both rolls; 223, slightly less stick but still splits; 213, sticky and splits; 214, excellent release, stays on front roll. All stocks have fair green strength.

(2) Mini-Die, 1° Arc , 100 RPM

TABLE VIII (CONTINUED)

MILL PROCESSING AND POST CURE STUDIES ON O-RING STOCKS

Compound R-193-	222	223	213	214
Stress-Strain				
Post Cure Study				
Press Cure (5 min. @ 370°F)				
10% M (psi)				
none	81	96	74	96
24 hr. @ 212°F	69	85	76	90
4 hr. @ 275°F	81	76	86	93
8 hr. @ " "	78	84	90	84
4 hr. @ 300°F	80	80	84	93
8 hr. @ " "	89	80	100	92
4 hr. @ 350°F	91	93	95	103
8 hr. @ " "	87	102	100	6
50% M (psi)				
none	210	342	334	343
24 hr. @ 212°F	164	279	293	307
4 hr. @ 275°F	195	208	323	332
8 hr. @ " "	197	238	335	285
4 hr. @ 300°F	186	211	344	318
8 hr. @ " "	208	218	382	322
4 hr. @ 350°F	237	267	361	321
8 hr. @ " "	225	300	351	304
100% M (psi)				
none	637	1195	1277	1037
24 hr. @ 212°F	509	1030	1146	1037
4 hr. @ 275°F	648	718	1227	1103
8 hr. @ " "	689	812	1217	1031
4 hr. @ 300°F	560	690	1292	1023
8 hr. @ " "	606	648	1364	1062
4 hr. @ 350°F	769	852	1251	998
8 hr. @ " "	663	939	1160	906
Tensile Strength (psi)				
none	1615	1796	1733	1621
24 hr. @ 212°F	1736	1389	1593	1561
4 hr. @ 275°F	1642	1788	1727	1592
8 hr. @ " "	1977	1671	1745	1545
4 hr. @ 300°F	1727	1683	1708	1506
8 hr. @ " "	1778	1705	1754	1536
4 hr. @ 350°F	1893	1682	1728	1594
8 hr. @ " "	1633	1697	1583	1585

TABLE VIII (CONTINUED)

MILL PROCESSING AND POST CURE STUDIES ON O-RING STOCKS

Compound R-193-	222	223	213	214
E_b (%)				
none	160	135	135	150
24 hr. @ 212°F	180	130	130	140
4 hr. @ 275°F	155	175	140	140
8 hr. @ " "	170	160	145	140
4 hr. @ 300°F	170	165	140	145
8 hr. @ " "	180	180	135	135
4 hr. @ 350°F	165	165	150	160
8 hr. @ " "	170	160	135	165
% Tension Set (@ Break)				
none	7	7	5	5
24 hr. @ 212°F	6	6	7	7
4 hr. @ 275°F	6	6	8	7
8 hr. @ " "	8	7	8	8
4 hr. @ 300°F	8	8	10	10
8 hr. @ " "	10	10	9	9
4 hr. @ 350°F	10	11	10	10
8 hr. @ " "	10	12	8	11
Shore A Hardness (3)				
none	52	54	54	52
24 hr. @ 212°F	43	50	52	50
4 hr. @ 275°F	47	50	52	52
8 hr. @ " "	50	50	51	49
4 hr. @ 300°F	47	50	51	52
8 hr. @ " "	50	50	56	51
4 hr. @ 350°F	49	51	55	55
8 hr. @ " "	51	54	54	53

(3) Test on stacked tensile strips.

TABLE VIII (CONTINUED)

MILL PROCESSING AND POST CURE STUDIES ON O-RING STOCKS

Compound R-193-	222	223	213	214
Compression Set (ASTM D-395, Method B) 70 hr. @ 275°F Press Cure - 5' @ 370°F				
Post Cure				
None	39	23	35	32
24 hr. @ 212°F	39	--	32	30
8 hr. @ 275°F	32	18	30	29
4 hr. @ 300°F	35	21	32	22
4 hr. @ 350°F	38	19	32	29
Tear Strength (ppi) (ASTM D-369, Die B) Press Cure - 8' @ 370°F Post Cure - 24 hr. @ 212°F	94	92	111	108
Aged Stress-Strain (336 hr. @ 300°F)				
10% M (psi)				
Post Cure				
None	128	120	121	120
24 hr. @ 212°F	135	--	106	120
8 hr. @ 275°F	127	104	109	126
4 hr. @ 300°F	128	127	116	122
4 hr. @ 350°F	125	124	110	117
50% M (psi)				
Post Cure				
None	329	339	367	327
24 hr. @ 212°F	341	--	329	355
8 hr. @ 275°F	318	278	330	370
4 hr. @ 300°F	309	356	342	334
4 hr. @ 350°F	322	336	323	329
100% M (psi)				
Post Cure				
None	815	892	879	742
24 hr. @ 212°F	844	--	830	793
8 hr. @ 275°F	777	698	853	818
4 hr. @ 300°F	749	933	869	749
4 hr. @ 350°F	741	856	771	724

TABLE VIII (CONTINUED)

MILL PROCESSING AND POST CURE STUDIES ON O-RING STOCKS

Compound R-193-	222	223	213	214
Aged Stress-Strain (contd.) 336 hr. @ 300°F				
Tensile Strength (psi)				
Post Cure				
None	1603	1525	1274	1221
24 hr. @ 212°F	1599	--	1273	1225
8 hr. @ 275°F	1576	1461	1280	1162
4 hr. @ 300°F	1393	1455	1220	1170
4 hr. @ 350°F	1572	1534	1236	1177
E_b (%)				
Post Cure				
None	170	170	165	175
24 hr. @ 212°F	170	--	160	165
8 hr. @ 275°F	180	180	160	150
4 hr. @ 300°F	160	150	150	160
4 hr. @ 350°F	190	165	165	175
% Tension Set (@ Break)				
Post Cure				
None	17	13	14	11
24 hr. @ 212°F	15	--	9	11
8 hr. @ 275°F	18	13	9	10
4 hr. @ 300°F	15	11	8	10
4 hr. @ 350°F	18	11	7	12
Aged 16 hr. @ 340°F (Steam)(4)				
10% M (psi)	395	325	154	172
50% M (psi)	498	488	400	402
100% M (psi)	656	736	813	763
Tensile Strength (psi)	995	964	813	763
E_b (%)	190	155	105	110
% Tension Set (@ Break)	75	54	15	16

(4) Same cure conditions as previous Stress-Strain.

TABLE VIII (CONTINUED)

MILL PROCESSING AND POST CURE STUDIES ON O-RING STOCKS

Compound R-193-	222	223	213	214
Fluid Resistance(5)				
70 hr. @ 73°F				
ASTM Fuel C				
% Wt. Change	4.54	6.37	3.32	7.02
% Vol. Swell	12.10	15.61	8.81	17.05
% Extracted	1.28	1.39	1.05	1.13
Mil-H-5606-C				
% Wt. Change	0.12	0.48	0.10	0.30
% Vol. Swell	0.40	1.09	0.60	0.62
% Extracted	0.18	0.07	0.16	0.12

(5) Press Cure - 8' @ 370°F
Post Cure - 24 hr. @ 212°F

TABLE IX

COMPOUNDING STUDIES TO IMPROVE HARDNESS AND TEAR STRENGTH OF CARBON BLACK-REINFORCED PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS

Compound R-193	-225	-226	-227	-218	-219
Polymer (K-17217)	100	100	100	100	100
SAF Black	5	10	5	5	5
FEF Black	10	10	10	10	10
Austin Black	20	20	30	20	--
Stan Mag ELC	6	6	6	6	6
Cab-O-lite P-4	--	--	--	--	20
Chem-Link 30	--	--	--	3	--
$(8\text{-}HQ)_2Zn$	1	1	1	1	1
Dicup 40C	3	2.75	2.75	2	2
Monsanto Rheometer Cure(1)					
@ 335°F					
Time to 2 pt. Rise (min.)	3.2	4.5	3.6	1.6	4.2
Time to Optimum Cure(min.)	15.3	22.3	15.3	17.8	15.3
Minimum Torque (ip)	7.0	8.0	7.0	7.2	8.0
Maximum Torque (ip)	15.7	14.8	14.7	16.5	15.6
Cure Rate Index	8.2	5.6	8.5	6.1	9.0
@ 370°F					
Time to 2 pt. Rise (min.)	1.6	1.6	1.6	0.9	1.1
Time to Optimum Cure(min.)	5.5	5.7	5.8	7.8	4.3
Minimum Torque (ip)	8.0	10.0	8.6	8.0	7.5
Maximum Torque (ip)	16.0	16.2	15.5	16.0	14.8
Cure Rate Index	25.3	24.4	23.8	14.5	31.2
Stress-Strain					
Press Cure (min. @ 370°F)	6	6	6	8	6
Post Cure (24 hr. @ 212°F)					
10% M (psi)	75	74	89	96	67
50% M (psi)	334	267	404	397	279
100% M (psi)	804	659	878	960	808
Tensile Strength (psi)	1234	1354	1228	1229	1359
E_b (%)	147	190	145	137	137
% Tension Set (@ Break)	4	7	6	2	8
Aged Stress-Strain					
Press Cure (min. @ 370°F	6	6	6	8	6
Post Cure (24 hr./212°F)					
10% M (psi)(Unaged)	75	74	89	96	67
240 hr. @ 275°F(Air)	117	126	130	136	96
672 hr. @ 275°F "	30	38	31	111	30
360 hr. @ 275°F (HF)(2)	30	35	33	51	29
672 hr. @ 275°F "	37	33	33	34	31
240 hr. @ 300°F (Air)	111	114	110	118	101
240 hr. @ 350°F "	56	59	55	83	78

TABLE IX (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE HARDNESS AND TEAR STRENGTH OF CARBON BLACK-REINFORCED PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS

Compound R-193	-225	-226	-227	-218	-219
Aged Stress-Strain					
50% M (psi) (Unaged)	334	267	404	397	279
240 hr. @ 275°F (Air)	448	380	470	504	317
672 hr. @ 275°F "	197	212	225	360	165
360 hr. @ 275°F (HF)(2)	215	173	209	266	185
672 hr. @ 275°F "	209	132	189	187	149
240 hr. @ 275°F (Air)	338	275	311	342	316
240 hr. @ 350°F "	127	82	107	146	174
100% M (psi)(Unaged)	804	659	878	960	808
240 hr. @ 275°F (Air)	889	738	845	1023	758
672 hr. @ 275°F "	502	451	515	703	442
360 hr. @ 275°F (HF)(2)	436	313	379	464	417
672 hr. @ 275°F "	397	224	328	332	298
240 hr. @ 275°F (Air)	586	453	504	597	717
240 hr. @ 350°F "	185	98	146	198	287
Tensile Strength (psi)(Unaged)	1234	1354	1228	1229	1359
240 hr. @ 275°F (Air)	1139	1135	1050	1191	1331
672 hr. @ 275°F "	949	808	772	918	1197
360 hr. @ 275°F (HF)(2)	760	557	578	584	948
672 hr. @ 275°F "	639	392	484	436	680
240 hr. @ 275°F (Air)	761	657	592	730	1167
240 hr. @ 350°F "	242	110	162	205	438
E_b (%) (Unaged)	147	190	145	137	137
240 hr. @ 275°F (Air)	135	177	140	132	150
672 hr. @ 275°F "	193	125	185	162	187
360 hr. @ 275°F (HF)(2)	177	202	180	150	167
672 hr. @ 275°F "	170	215	182	172	193
240 hr. @ 300°F (Air)	147	185	143	147	152
240 hr. @ 350°F "	170	175	167	142	180
% Tension Set (@ Break)(Unaged)	4	7	6	2	8
240 hr. @ 275°F (Air)	5	7	9	2	9
672 hr. @ 275°F "	5	7	5	3	12
360 hr. @ 275°F (HF)(2)	6	11	7	7	10
672 hr. @ 275°F "	--	--	--	--	--
240 hr. @ 300°F (Air)	4	7	4	4	11
240 hr. @ 350°F "	15	26	12	7	13

TABLE IX (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE HARDNESS AND TEAR STRENGTH OF CARBON BLACK-REINFORCED PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS

Compound R-193	-225	-226	-227	-218	-219
Shore A Hardness					
Press Cure - 16' @ 370°F					
Post Cure - 24 hr. @ 212°F					
Unaged	51	52	53	53	44
240 hr. @ 275°F (Air)	53	55	58	56	45
672 hr. @ 275°F "	55	58	59	57	49
360 hr. @ 275°F (HF)(2)	48	49	49	53	53
672 hr. @ 275°F "	49	48	51	53	40
240 hr. @ 300°F "	55	55	55	57	43
240 hr. @ 350°F "	45	47	50	52	40
% Compression Set(3)(4) (70 hr. @ 275°F)	32	54	39	25	33
Tear Strength (ppi)(@ 73°F)(4) (ASTM D-369, Die B)	74	112	79	76	88
Abrasive Index(4)(5) (ASTM D-1630)	59	84	57	97	100
Fluid Resistance					
70 hr. @ 73°F					
ASTM Fuel C					
% Wt. Change	3.30	3.48	3.50	3.17	2.46
% Vol. Swell	9.15	8.40	7.70	7.80	7.49
% Extracted	1.15	1.18	1.00	1.10	1.35
Mil-H-5606-C					
% Wt. Change	0.00	0.02	0.00	-0.02	0.00
% Vol. Swell	-0.16	-0.04	0.37	0.00	0.04
% Extracted	0.16	0.18	0.00	0.15	0.18
240 hr. @ 275°F					
Mil-H-5606-C					
% Wt. Change	-0.70	-0.38	-0.43	-1.30	-2.50
% Vol. Swell	1.16	1.96	1.54	-0.04	0.33
% Extracted	0.96	0.64	0.67	1.33	1.67

TABLE IX (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE HARDNESS AND TEAR STRENGTH OF CARBON BLACK-REINFORCED PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS

Compound R-193	-225	-226	-227	-218	-219
Young's Bending Modulus (psi)					
@ 20°C	861	894	801	863	568
0	707	901	746	1001	607
-20	776	1048	801	1196	665
-30	896	1341	845	1359	712
-40	1205	2313	1803	1933	1118
-50	1879	3245	2496	2990	1619
-60	7458	12039	9771	10466	6215
-67	78892	109312	105777	98688	86095
Recovery (psi)	776	1048	801	1196	665
YMI (°C)	-62	-59	-61	-60	-63

(1) Mini-Die, 1° Arc, 100 rpm
(2) Hydraulic Fluid - Mil-H-5606-C
(3) ASTM D-395, Method B, 25% Deflection
(4) Press Cure - 12' @ 370°F, Post Cure - 24 hr. @ 212°F
(5) Test run on YMI specimens

TABLE X

PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS SUBMITTED TO PARKER SEAL FOR EVALUATION IN SECOND QUARTER

Compound R-193	-228	-229
Polymer (K-17217)	100	100
Quso WR 82	30	30
Stan Mag ELC	6	6
Stabilizer *	2	2
Silastic 410	--	15
Dicup 40C	1	1
Rubber Mill Processing @ 130°F		
Release	Poor	Fair
Split to both rolls	Yes	Yes but better
Green Strength	Good	Good
Nerve (smoothness)	Fair	Good
Stress-Strain (Dumbbell)(0.040" thick)		
Press Cure - 15' @ 370°F		
Post Cure - 4 hr. @ 350°F		
50% M (psi)	457	692
100% M (psi)	1248	1367
Tensile Strength (psi)	1609	1497
E_b (%)	143	115
% Tension Set (@ Break)	10	7
Shore A Hardness(1)	55	70
Compression Set(1) (70 hr. @ 275°F)		
Cylinder	28	24
Plied Disk	35	32
Tear Strength (ppi)(Die B)	112	107
NBS Abrasive Index	110	--

* Bis(8-hydroxyquinoline Zinc)II

TABLE X (CONTINUED)

PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS SUBMITTED TO PARKER SEAL FOR EVALUATION IN SECOND QUARTER

Compound R-193	-228	-229	
Young's Modulus in Flexure (psi) (1)			
@ 20°C	918	--	
0	983	--	
-20	1139	--	
-30	1335	--	
-40	1835	--	
-50	3671	--	
-60	13218	--	
-67	107707	--	
Recovery (psi)	1180	--	
YMI (°C)	-59	--	
Shore A Hardness (Unaged)	55	--	
Aged 240 hr. @ 275°F (Air)	62	--	
672 hr. @ 275°F "	60	--	
672 hr. @ 300°F "	62	--	
336 hr. @ 350°F "	62	--	
360 hr. @ 275°F (HF) (2)	55	--	
672 hr. @ 275°F "	53	--	
Aged Stress-Strain (Cut-Ring)			
Rings cut from 6"x6"x0.075" slabs			
Press Cure - 15' @ 370°F			Dumbbell (cut
Post Cure - 4 hr. @ 350°F			from same slab)
50% M (psi)(Unaged)	104	228	355
Aged 240 hr. @ 275°F (Air)	169	307	
672 hr. @ 275°F "	139	--	
360 hr. @ 275°F (HF) (2)	118	234	
672 hr. @ 275°F "	88	--	
240 hr. @ 300°F (Air)	--	322	
672 hr. @ 300°F "	175	--	
240 hr. @ 350°F "	--	309	
336 hr. @ 350°F "	152	--	
100% M (psi)(Unaged)	329	478	759
Aged 240 hr. @ 275°F (Air)	507	630	
672 hr. @ 275°F "	451	--	
360 hr. @ 275°F (HF) (2)	339	447	
672 hr. @ 275°F "	253	--	
240 hr. @ 300°F (Air)	--	577	
672 hr. @ 300°F "	453	--	
240 hr. @ 350°F "	--	619	
336 hr. @ 350°F "	434	--	

TABLE X (CONTINUED)

PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS
SUBMITTED TO PARKER SEAL FOR EVALUATION IN SECOND QUARTER

Compound R-193	-228	-229	Dumbbell (cut from same slab)
Aged Stress-Strain			
Tensile Strength (psi)(Unaged)	1109	900	1344
Aged 240 hr. @ 275°F (Air)	1115	1028	
672 hr. @ 275°F "	1076	--	
360 hr. @ 275°F (HF)[2]	1063	751	
672 hr. @ 275°F "	842	--	
240 hr. @ 300°F (Air)	--	758	
672 hr. @ 300°F "	849	--	
240 hr. @ 350°F "	--	986	
336 hr. @ 350°F "	1034	--	
E_b (%) (Unaged)	220	179	180
Aged 240 hr. @ 275°F (Air)	200	176	
672 hr. @ 275°F "	204	--	
360 hr. @ 275°F (HF)[2]	248	191	
672 hr. @ 275°F "	292	--	
240 hr. @ 300°F (Air)	--	159	
672 hr. @ 300°F "	200	--	
240 hr. @ 350°F "	--	175	
336 hr. @ 350°F "	229	--	
Fluid Resistance[1]			
70 hr. @ 73°F			
ASTM Fuel C			
% Wt. Change	4.61	8.82	
% Vol. Swell	12.53	20.57	
% Extracted	1.38	1.62	
Mil-H-5606-C			
% Wt. Change	0.18	2.97	
% Vol. Swell	0.53	6.15	
% Extracted	0.21	0.14	

(1) Press Cure - 15' @ 370°F, Post Cure - 4 hr. @ 350°F
(2) HF - Hydraulic Fluid Mil-H-5606-C

TABLE XI

PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUND SENT TO THE ARMY (WATERTOWN) FOR ENVIRONMENTAL TESTING

Compound	R-193234
Polymer (K-17638)	100
Quso WR 82	30
Stan Mag ELC	6
Stabilizer(1)	2
Dicup 40C	0.75

Mixing 10 Brabender mixes of masterbatch were made. The stabilizer and peroxide were then added to the masterbatch on a mill to give 1387 g. of compound.

Stress-Strain						
Press Cure (min. @ 320°F)		45			60	
Post Cure (24 hr. @ 212°F)						
Slab Thickness (in.)		0.050			0.075	
Test Specimen(2)	DB,WG	DB,AG	R	DB,WG	DB,AG	R
50% M (psi)	258	216	193	279	268	161
100% M (psi)	628	489	517	721	598	438
Tensile Strength (psi)	1161	925	1150	1157	1040	1175
E_b (%)	180	197	210	175	195	227
% Tension Set (@ Break)	11	12	--	7	14	--

Shore A Hardness	65
Compression Set	
70 hr. @ 275°F	34
300°F	38
325°F	39
NBS Abrasive Index	88
Young's Modulus in Flexure	
@ 20°C	1110
0	1547
-20	1654
-30	1713
-40	3108
-50	4724
-60	11242
-67	75376
Recovery (psi)	1635
YMI (°C)	-60

(1) Bis(8-hydroxyquinoline Zinc)(II)

(2) DB = Dumbbell; WG = with mill grain; AG = against mill grain; R = cut ring.

TABLE XI (CONTINUED)

PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUND SENT TO THE ARMY (WATERTOWN) FOR ENVIRONMENTAL TESTING

Compound	R-193234	
Stress-Strain		
Cut from 0.075" thick slabs	Ring	Dumbbell
Press Cure - 60' @ 320°F		
Post Cure - 34 hr. @ 212°F		
50% M (psi)(Unaged)	161	279
240 hr. @ 300°F	239	462
336 hr. @ 300°F	226	369
240 hr. @ 350°F	241	356
336 hr. @ 350°F	201	259
100% M (psi)(Unaged)	438	721
240 hr. @ 300°F	614	978
336 hr. @ 300°F	605	805
240 hr. @ 350°F	519	700
336 hr. @ 350°F	414	451
Tensile Strength (psi)(Unaged)	1175	1157
240 hr. @ 300°F	1159	1236
336 hr. @ 300°F	1052	1110
240 hr. @ 350°F	778	812
336 hr. @ 350°F	597	595
E_b (%) (Unaged)	227	175
240 hr. @ 300°F	213	150
336 hr. @ 300°F	192	170
240 hr. @ 350°F	173	135
336 hr. @ 350°F	166	170
% Tension Set (@ Break)(Unaged)	--	7
240 hr. @ 300°F	--	11
336 hr. @ 300°F	--	--
240 hr. @ 350°F	--	8
336 hr. @ 350°F	--	23

TABLE XII

COMPOUNDING STUDIES TO IMPROVE CUT-RING STRESS-STRAIN PROPERTIES

Compound K-193	-235			-236			-237			-238			-239		
Polymer (K-17438)	100			100			100			100			100		
Quso WR 82	--			20			25			30			30		
Stan Mag ELC	6			6			6			6			6		
Stabilizer[(1)]	2			2			2			2			2		
Dicup 40C	1			1			1			1			2		
Stress-Strain[(2)]		DB[(4)]			DB			DB			DB			DB	
	R[(3)]	WG	AG	R	WG	AG	R	WG	AG	R	WG	AG	R	WG	AG
Press Cure - 60' @ 320°F															
Post Cure - 4 hr. @ 350°F															
50% M (psi)	28	61	73	151	389	256	207	444	322	286	550	429	486	796	730
100% M (psi)	85	185	224	622	1103	679	717	1231	778	839	1111	831	--	--	--
Tensile Strength (psi)	274	311	289	1262	1300	1001	1398	1517	1147	1238	1247	1057	1285	1239	1087
E_b (%)	161	130	135	150	120	160	161	130	155	151	135	160	105	100	100
% Tension Set (@ break)	--	2	1	--	4	7	--	6	10	--	12	13	--	11	9
Shore A Hardness	30			57			65			72			75		

(1) Bis(8-hydroxyquinoline Zinc)(II)
(2) Specimens cut from 0.075" thick slabs
(3) R = cut ring; DB = dumbbell; WG = with mill grain; AG = against mill grain

TABLE XIII

EVALUATION OF QUSO G-32 TREATED WITH A SILANE COUPLING AGENT (DOW CORNING A-174)

Compound R-193	-255	-256	-257
Polymer (K-17638)	100	100	100
Quso G-32	30	--	--
Quso G-32 (Silane-Treated)(1)	--	30	30
Stan-Mag ELC	6	6	6
$(8\text{-HQ})_2Zn$ (Stabilizer)	2	2	--
Dicup 40C	1	1	1
Stress-Strain			
Press Cure - 30' @ 320°F			
Post Cure - 4 hr. @ 350°F			
10% M (psi)	163	180	214
50% M (psi)	452	432	529
100% M (psi)	839	732	928
Tensile Strength(psi)	991	858	956
E_b (%)	140	135	105
% Tension Set (@ Break)	18	8	8
Shore A Hardness(2)	73	70	73
Compression Set (%)(2)			
70 hr. @ 275°F	75	71	83
Tear Strength (ppi)(Die B)(2)			
@ 73°F	93	98	89
NBS Abrasive Index	33	41	39
Aged Stress-Strain			
Press Cure - 30' @ 320°F			
Post Cure - 4 hr. @ 350°F			
10% M (psi)(Unaged)	100	94	133
Aged 240 hr. @ 300°F	112	117	127
48 hr. @ 350°F	57	58	70
240 hr. @ 350°F	89	109	120
336 hr. @ 350°F	141	119	168
50% M (psi)(Unaged)	510	492	511
Aged 240 hr. @ 300°F	553	475	465
48 hr. @ 350°F	439	335	290
240 hr. @ 350°F	336	320	--
336 hr. @ 350°F	311	295	--

TABLE XIII (CONTINUED)

EVALUATION OF QUSO G-32 TREATED WITH A SILANE COUPLING AGENT (DOW CORNING A-174)

Compound R-194	-255	-256	-257
Aged Stress-Strain			
100% M (psi)(Unaged)	1001	810	737
Aged 240 hr. @ 300°F	895	--	--
48 hr. @ 350°F	819	544	430
240 hr. @ 350°F	--	--	--
336 hr. @ 350°F	--	--	--
Tensile Strength (psi)(Unaged)	1175	876	772
Aged 240 hr. @ 300°F	895	625	625
48 hr. @ 350°F	839	576	458
240 hr. @ 350°F	463	389	267
336 hr. @ 350°F	321	326	246
E_b (%) (Unaged)	130	125	115
Aged 240 hr. @ 300°F	100	100	105
48 hr. @ 300°F	110	125	125
240 hr. @ 300°F	95	95	45
336 hr. @ 300°F	60	85	40
% Tension Set (@ Break)(Unaged)	12	7	10
Aged 240 hr. @ 300°F	7	8	12
48 hr. @ 350°F	8	8	13
240 hr. @ 350°F	12	12	15
336 hr. @ 350°F	14	12	16
Young's Bending Modulus (psi) @ 20°C	2153	2455	2885

(1) Quso G-32 (100 g) was treated with Union Carbide Silane (A-174) (20.0 g, 19.2 ml) in methyl alcohol (1000 ml) (pH adjusted to 3 with glacial acetic acid). The reaction mixture was stirred for 16 hr. @ 73°F. The silica was isolated by filtration on a Büchner funnel, washed with methanol and hexane and then dried 5 hrs. @ 70°C in a vacuum oven.

(2) Press Cure 60' @ 320°F, Post Cure 4 hr. @ 350°F

TABLE XIV

COMPOUNDING STUDIES TO IMPROVE STRESS-STRAIN PROPERTIES OF CUT-RING SPECIMENS

Compound R-193-	259	260	261	262	263	264
Polymer (K-17638)	100	100	100	100	100	100
Quso WR-82	25	30	--	25	--	--
FEF Black	--	--	25	--	25	--
Cab-O-lite P-4	--	--	--	30	30	50
Stan Mag ELC	6	6	6	6	6	6
$(8\text{-HQ})_2Zn$ (Stabilizer)	2	2	2	2	2	2
Dicup 40C	2	2	2	2	2	2

Rubber Mill Processing -- All compounds have low green strength, stick to mill rolls and have generally poor processing characteristics.

Monsanto Rheometer Cure (1)						
@ 335°F						
Time to 2 pt. Rise (min.)	1.7	1.6	2.4	1.8	2.1	1.5
Time to Optimum Cure (min.)	11.0	13.0	12.5	10.5	10.8	9.8
Minimum Torque (ip)	7.4	8.0	8.3	7.9	8.9	6.8
Maximum Torque (ip)	24.2	27.5	21.5	26.6	22.8	18.2
Cure Rate Index	10.7	8.8	9.9	11.5	11.5	12.0
@ 370°F						
Time to 2 pt. Rise (min.)	1.5	0.5	1.0	0.8	0.8	1.0
Time to Optimum Cure (min.)	9.5	5.3	3.5	4.0	3.5	2.7
Minimum Torque (ip)	4.8	9.0	9.0	8.0	9.0	7.3
Maximum Torque (ip)	10.8	28.4	22.0	27.3	23.0	17.7
Cure Rate Index	5.6	20.8	40.0	31.2	37.0	58.8

(1) Mini Die, 100 RPM, 1° Arc

TABLE XIV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE STRESS-STRAIN PROPERTIES OF CUT-RING SPECIMENS

Compound R-193-	259		260		261		262		263		264	
Stress-Strain	DB[2]	R[3]	DB	R	DB	R	DB	R	DB	R	DB	R
Press Cure - 30' @ 320°F												
Post Cure - 4 hrs. @ 350°F												
50% M (psi)	446	207	630	268	450	176	786	211	806	284	523	187
100% M (psi)	1442	926	--	1023	1607	745	1382	716	--	657	--	442
Tensile Strength (psi)	1442	1211	1278	1290	1694	1410	1386	991	1378	1018	726	657
E_b (%)	100	136	85	126	115	163	100	140	95	156	80	146
% Tension Set (@ Break)	6	--	7	--	--	--	14	--	10	--	10	--
Shore A Hardness	60		66		56		68		66		49	
Press Cure - min. @ 370°F	40		10		10		10		10		10	
Post Cure - 4 hrs. @ 350°F												
% Compression Set[4]												
70 hr. @ 275°F	21		25		23		35		28		21	
70 hr. @ 300°F	25		28		27		37		31		20	
70 hr. @ 350°F	--		52		55		--		--		--	
138 hr. @ 350°F	48		--		--		68		73		60	
Tear Strength (Die B)(ppi)[4]	161		204		243		135		169		87	
NBS Abrasive Index[5]	30		33		57		22		27		26	

(2) DB = Dumbbell Specimen
(3) R = Cut-Ring Specimen
(4) Same cure conditions as for Shore A Hardness specimens.
(5) Run on YMI block.

TABLE XIV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE STRESS-STRAIN PROPERTIES OF CUT-RING SPECIMENS

Compound R-193-	259		260		261		262		263		264	
Aged Stress-Strain	DB	R	DB	R	DB	R	DB	R	DB	R	DB	R
Press Cure - 10 min. @ 370°F												
Post Cure - 4 hr. @ 350°F												
50% M(psi) (Unaged)	621	205	727	287	470	214	644	338	603	274	373	121
Aged 240 hr. @ 275°F (Air)		290		386		237		--		--		--
336 hr. @ " " "		--		--		--		487		368		168
672 hr. @ " " "		308		406		246		--		--		--
1000 hr. @ " " "		380		444		249		--		--		--
241.5 hr. @ " " (HF)		224		184		289		--		--		--
360.0 hr. @ " " "		--		--		--		347		220		121
696.5 hr. @ " " "		250		317		153		376		200		156
1000 hr. @ " " "		190		303		138		307		161		122
240 hr. @ 300°F (Air)		294		375		232		--		--		--
336 hr. @ " " "		--		--		--		472		380		185
672 hr. @ " " "		256		372		210		--		--		--
1000 hr. @ " " "		--		337		242		--		--		--
240 hr. @ 350°F "		272		371		185		--		--		--
336 hr. @ " " "		--		--		--		387		251		145
672 hr. @ " " "		177		231		117		--		--		--
1000 hr. @ " " "		--		--		88		--		--		--
240 hr. @ 400°F "		158		--		79		--		--		--
336 hr. @ " " "		--		--		--		D[6]		D		D
672 hr. @ " " "		D[6]		D		D		--		--		--

(6) Degraded - No Test

TABLE XIV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE STRESS-STRAIN PROPERTIES OF CUT-RING SPECIMENS

Compound R-193-	259		260		261		262		263		264	
Aged Stress-Strain (contd.)	DB	R	DB	R	DB	R	DB	R	DB	R	DB	R
100% M (psi) (Unaged)	--	905	--	935	--	840	1238	710	1132	615	--	363
Aged 240 hr. @ 275°F (Air)		987		1042		858		--		--		--
336 hr. @ " " "		--		--		--		845		707		414
672 hr. @ " " "		900		1001		818		--		--		--
1000 hr. @ " " "		--		996		789		--		--		--
241.5 hr. @ 275°F (HF)		694		540		814		--		--		--
360 hr. @ " " "		--		--		--		605		380		261
696.5 hr. @ " " "		631		727		367		554		308		255
1000 hr. @ " " "		504		671		288		450		242		207
240 hr. @ 300°F (Air)		959		977		760		--		--		--
336 hr. @ " " "		--		--		--		789		687		427
672 hr. @ " " "		826		924		650		--		--		--
1000 hr. @ " " "		889		850		638		--		--		--
240 hr. @ 350°F "		700		818		407		--		--		--
336 hr. @ " " "		--		--		--		--		--		253
672 hr. @ " " "		269		--		175		--		--		--
1000 hr. @ " " "		--		--		103		--		--		--
240 hr. @ 400°F "		--		--		90		--		--		--
336 hr. @ " " "		--		--		--		D(6)		D		D
672 hr. @ " " "		D(6)		D		D		--		--		--
Tensile Strength (psi)(Unaged)	1218	1245	1359	1287	1005	1167	1178	1038	1259	965	603	503
Aged 240 hr. @ 275°F (Air)		1179		1256		1246		--		--		--
336 hr. @ " " "		--		--		--		952		935		520
672 hr. @ " " "		1076		1089		1266		--		--		--
1000 hr. @ " " "		1123		1165		1240		--		--		--
241.5 hr. @ 275°F (HF)		1174		1005		1217		--		--		--
360 hr. @ " " "		--		--		--		807		547		318
696.5 hr. @ " " "		977		1010		674		634		362		305
1000 hr. @ " " "		831		901		509		502		279		240

TABLE XIV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE STRESS-STRAIN PROPERTIES OF CUT-RING SPECIMENS

Compound R-193-	259		260		261		262		263		264	
Aged Stress-Strain (contd.)	DB	R	DB	R	DB	R	DB	R	DB	R	DB	R
Tensile Strength(psi)(Unaged)												
Aged 240 hr. @ 300°F (Air)		1146		1138		1160		--		--		--
336 hr. @ " " "		--		--		--		841		843		555
672 hr. @ " " "		1027		1079		1037		--		--		--
1000 hr. @ " " "		1002		893		936		--		--		--
240 hr. @ 350°F "		897		914		527		--		--		--
336 hr. @ " " "		--		--		--		527		342		268
672 hr. @ " " "		306		317		193		--		--		--
1000 hr. @ " " "		191		220		103		--		--		--
240 hr. @ 400°F "		158		210		90		--		--		--
336 hr. @ " " "		--		--		--		D		D		D
672 hr. @ " " "		D		D		D		--		--		--
E_b (%) (Unaged)	76	124	87	134	107	126	103	145	117	160	93	139
Aged 240 hr. @ 275°F (Air)		119		125		137		--		--		--
336 hr. @ " " "		--		--		--		120		147		130
672 hr. @ " " "		117		110		147		--		--		--
1000 hr. @ " " "		125		125		153		--		--		--
241.5 hr. @ " " (HF)		147		170		143		--		--		--
360 hr. @ " " "		--		--		--		138		173		160
696.5 hr. @ " " "		175		155		202		136		160		160
1000 hr. @ " " "		160		142		200		125		150		145
240 hr. @ 300°F (Air)		116		120		145		--		--		--
336 hr. @ " " "		--		--		--		110		137		140
672 hr. @ " " "		120		120		153		--		--		--
1000 hr. @ " " "		125		107		153		--		--		--
240 hr. @ 350°F "		131		117		140		--		--		--
336 hr. @ " " "		--		--		--		90		92		120
672 hr. @ " " "		135		100		163		--		--		--
1000 hr. @ " " "		45		20		160		--		--		--
240 hr. @ 400°F "		57		20		127		--		--		--
336 hr. @ " " "		--		--		--		D		D		D
672 hr. @ " " "		D		D		D		--		--		--

TABLE XIV (CONTINUED)

COMPOUNDING STUDIES TO IMPROVE STRESS-STRAIN PROPERTIES OF CUT-RING SPECIMENS

Compound R-193-	259	260	261	262	263	264
Young's Bending Modulus @ RT (20°C)	1065	1081	855	1497	1310	895

TABLE XV

EVALUATION OF SELECTED VULCANIZING AGENTS FOR PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS

Compound R-193	-265		-266		-267		-268		-269	
Polymer (K-1[illegible]638)	100		100		100		100		100	
Quso WR 82	30		30		30		30		30	
Stan Mag ELC	6		6		--		6		6	
$(8\text{-HQ})_2Zn$ (Stabilizer)	2		2		2		2		2	
Dicup R	0.8		--		--		--		--	
Dicup 40C	--		2		2		--		--	
Cadox BS	--		--		--		1.44		--	
Cadox TS-50	--		--		--		--		1.84	
Rubber Mill Processing	All compounds have low green strength and stick to rolls.									
Monsanto Rheometer Cure[1]										
@ °F	340		340		340		250		212	
Time to 2 pt. Rise (min.)	1.8		1.7		1.8		"no cure"		4.6	
Time to Optimum Cure (min.)	10.0		11.0		11.1				5.8	
Minimum Toruqe (ip)	9.0		8.8		8.0				1.2	
Maximum Torque (ip)	25.0		26.0		21.2				3.6	
Cure Rate Index	12.2		10.8		10.8				83.3	
Stress-Strain	DB	R	DB	R	DB	R	DB[2]	R	DB[2]	R
Press Cure - min/°F	15/340		15/340		15/340		13/250		17/212	
Post Cure - 4 hr. @ 350°F										
50% M (psi)	1003	357	964	426	741	338	60	"no	48	50
100% M (psi)	--	1135	--	1206	--	1035	79	test"	55	62
Tensile Strength (psi)	1381	1386	1254	1426	1134	1433	101		73	87
E_b (%)	75	115	75	117	75	120	280		235	215
% Tension Set (@ Break)	7	--	7	--	7	--	30		23	--
Shore A Hardness	66		66		58		"no test"		"no test"	
Press Cure - 20' @ 340°F										
Post Cure - 4 hr. @ 350°F										
% Compression Set[3] (70 hr. @ 275°F)	25		24		21		"no test"		"no test"	

TABLE XV (CONTINUED)

EVALUATION OF SELECTED VULCANIZING AGENTS FOR PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS

Compound R-193	-265		-266		-267		-268	-269
Aged Stress-Strain	DB	R	DB	R	DB	R	"no test"	"no test"
Press Cure - 10' @ 340°F								
Post Cure - 4 hr. @ 350°F								
50% M (psi)(Unaged)	837	286	830	296	785	206		
Aged 240 hr. @ 300°F	864	413	792	418	596	278		
240 hr. @ 350°F	598	370	654	392	424	229		
100% M (psi)(Unaged)	--	979	--	991	--	812		
Aged 240 hr. @ 300°F	--	1134	--	1089	--	853		
240 hr. @ 350°F	996	888	--	909	804	583		
Tensile Strength (psi)(Unaged)	1324	1223	1402	1252	1176	1169		
Aged 240 hr. @ 300°F	1221	1343	1301	1255	1207	1117		
240 hr. @ 350°F	996	985	1008	995	860	830		
E_b (%) (Unaged)	85	120	90	132	80	129		
Aged 240 hr. @ 300°F	70	120	85	120	100	130		
240 hr. @ 350°F	100	113	80	112	110	140		
% Tension Set (@ Break)(Unaged)	8	--	9	--	7	--		
Aged 240 hr. @ 300°F	9	--	8	--	7	--		
240 hr. @ 350°F	14	--	15	--	15	--		

(1) Mini-Die, 1° Arc, 100 rpm
(2) Poor cure, specimens are blistered and soft.
(3) Same cure conditions as for Shore A Hardness.

TABLE XVI

EVALUATION OF MORE SELECTED VULCANIZING AGENTS FOR PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS

Compound R-193	-270	-271	-272	-273	-274	-275
Polymer (K-17638)	100	100	100	100	100	100
Quso WR82	30	30	30	30	30	30
Stan Mag ELC	6	6	6	6	6	6
$(8\text{-HQ})_2Zn$ (Stabilizer)	2	2	2	2	2	2
Vulcup R	0.5	--	--	--	--	--
Varox	--	0.86	--	--	--	--
Percadox 29/40	--	--	1.12	--	--	--
Luperco 130 XL	--	--	--	0.94	--	--
Luperco 230 XL	--	--	--	--	1.27	--
Di-t-Butyl Peroxide	--	--	--	--	--	0.43

Rubber Mill Processing All compounds have poor green strength and stick to mill rolls.

Monsanto Rheometer Cure[1]						
@ °F	340	320	340	340	320	340
Time to 2 pt. Rise (min.)	2.2	4.5	2.3	18.5	2.8	27.3
Time to Optimum Cure (min.)	22.0	39.5	12.5	45.0	20.5	27.3
Minimum Torque (ip)	7.4	7.0	7.3	6.8	7.4	6.0
Maximum Torque (ip)	24.5	18.4	10.9	9.8	18.2	8.0
Cure Rate Index	5.0	2.8	9.8	3.7	5.6	0.0

Stress-Strain	DB	R	DB	R	DB	R	DB	R	DB	R	DB	R
Press Cure - min. @ °F	22/340		34/320		5/340		42/340		8/340		42/340	
Post Cure - 4 hr. @ 350°F												
50% M (psi)	1231	337	649	282	320	123	145	90	552	214	109	75
100% M (psi)	--	1256	1355	790	877	273	315	199	1176	642	222	185
Tensile Strength (psi)	1339	1266	1492	1253	1395	1268	977	908	1310	1130	850	826
E_b (%)	65	103	120	157	175	297	320	435	125	168	410	477
% Tension Set (@ Break)	5	--	10	--	13	--	30	--	8	--	38	--

TABLE XVI (CONTINUED)

EVALUATION OF MORE SELECTED VULCANIZING AGENTS FOR PHOSPHONITRILIC FLUOROELASTOMER O-RING COMPOUNDS

Compound R-193	-270		-271		-272		-273		-274		-275	
Shore A Hardness	71		62		47		45		60		35	
Press Cure (min. @ °F)												
Post Cure (4 hr. @ 350°F)												
% Compression Set(1)												
(70 hr. @ 275°F)	18		23		40		62		33		100	
Aged Stress-Strain	DB	R	DB	R	DB	R	DB	R	DB	R	DB	R
Press Cure - min. °F												
Post Cure - 4 hr. @ 350°F												
50% M (psi)(Unaged)	994	375	537	271	282	96	155	80	506	230	114	58
Aged 240 hr. @ 300°F	868	570	406	292	186	133	134	222	364	285	163	78
240 hr. @ 350°F	829	462	446	277	235	133	183	116	404	275	122	86
100% M (psi)(Unaged)	--	1265	1165	777	753	278	356	172	1079	609	201	98
Aged 240 hr. @ 300°F	--	1282	954	823	469	372	286	321	801	711	335	151
240 hr. @ 350°F	--	1066	776	639	413	305	284	233	609	570	173	143
Tensile Strength (psi)(Unaged)	1552	1384	1321	1349	1401	1325	1058	874	1322	1234	473	337
Aged 240 hr. @ 300°F	1310	1282	1258	1234	1172	1037	830	778	1137	1078	439	421
240 hr. @ 350°F	1032	1079	814	875	530	572	335	393	632	693	225	287
E_b (%) (Unaged)	85	115	130	167	205	320	360	430	145	197	380	575
Aged 240 hr. @ 300°F	75	100	145	159	255	280	340	321	175	172	350	380
240 hr. @ 350°F	95	102	115	150	165	220	175	220	135	137	200	280
% Tension Set (@ Break)(Unaged)	8	--	12	--	14	--	38	--	10	--	45	--
Aged 240 hr. @ 300°F	10	--	14	--	22	--	32	--	19	--	36	--
240 hr. @ 350°F	12	--	16	--	23	--	18	--	18	--	27	--

(1) Mini-Die, 1° Arc, 100 rpm
(2) Same cure conditions as for Shore A Hardness

TABLE XVII

O-RING COMPOUND FOR DUMBBELL VERSUS CUT-RING STRESS-STRAIN MEASUREMENTS

	R-193276
Polymer (K-17638)	100
Quso WR 82	25
Stan Mag ELC	6
$(8\text{-HQ})_2$Zn (Stabilizer)	1
Dicup 40 C	1

4 - 6"x6"x0.075" slabs were press cured 60' @ 320°F and post-cured 4 hrs. @ 350°F in a forced-air oven.

TABLE XVII-A

DUMBBELL VERSUS CUT-RING STRESS-STRAIN MEASUREMENTS ON O-RING COMPOUND R-193276

Slab No.	Specimen No.	No. of Tests (n)	Cut at	Test at	50% $\overline{M}$[1] (psi) C[9]	50% $\overline{M}$[1] (psi) B[10]	100% $\overline{M}$[1] (psi) C	100% $\overline{M}$[1] (psi) B	$\overline{T}$[2] (psi) C	$\overline{T}$[2] (psi) B	$\overline{E}_b$[3] (%) C	$\overline{E}_b$[3] (%) B
1	DB[7]-1 to 3	3	F[5]	F	143	186	537	604	1273	1314	190	188
		s[4] (psi)			28	41	103	125	42	61	20	27
		CV[11] (psi)			19.5	22.0	19.2	20.7	3.2	4.6	11	14
"	R[8]-1 to 17	17	F	F	94	148	463	630	1342	1372	179	166
		s (psi)			4	15	18	30	111	111	11	9
		CV (%)			4.3	10.1	3.9	4.8	8.3	8.0	6.1	5.4
2	DB-1 to 3	3	F	F	149	206	787	928	1524	1556	150	140
		s (psi)			10	14	61	77	149	97	0	0
		CV (%)			6.7	6.8	7.8	8.3	9.8	6.2	--	--
"	R-1 to 10	10	F	F	95	177	495	701	1286	1303	166	149
		s (psi)			4	19	24	40	133	128	7	9
		CV (%)			4.2	10.7	4.8	5.7	10.3	9.8	4.2	6.0
3	R-1 to 12-A	12	F	F	95	183	518	731	1435	1444	172	161
		s (psi)			4	20	29	30	61	67	7	7
		CV (%)			4.2	10.9	5.6	4.1	4.2	4.6	4.1	4.3
"	R-1 to 13-B	13	F	H[6]	--	168	--	740	--	1470	--	155
		s (psi)			--	12	--	50	--	93	--	8
		CV (%)			--	7.1	--	6.8	--	6.3	--	5.2
4	R-1 to 15-A	15	H	F	89	147	482	630	1343	1372	175	168
		s (psi)			5	17	42	60	111	103	10	10
		CV (%)			5.6	11.6	8.7	9.5	8.3	7.5	5.7	6.0
"	R-1 to 17-B	17	H	H	--	142	--	663	--	1427	--	161
		s (psi)			--	26	--	55	--	94	--	9
		CV (%)			--	18.3	--	8.3	--	6.6	--	5.6

TABLE XVII-A (CONTINUED)

DUMBBELL VERSUS CUT-RING STRESS-STRAIN MEASUREMENTS ON O-RING COMPOUND R-193276

Slab No.	Specimen No.	No. of Tests (n)	Cut at	Test at	50% $\overline{M}$[1] (psi) C[9]	50% $\overline{M}$[1] (psi) B[10]	100% $\overline{M}$[1] (psi) C	100% $\overline{M}$[1] (psi) B	$\overline{T}$[2] (psi) C	$\overline{T}$[2] (psi) B	$\overline{E}_b$[3] (%) C	$\overline{E}_b$[3] (%) B
	DB (total)	6			--	196	--	765	--	1435	--	164
		s (psi)			--	29	--	200	--	151	--	32
		CV (%)			--	14.8	--	26.1	--	10.5	--	19.5
	R (total)	84			--	158	--	676	--	1399	--	161
		s (psi)			--	24	--	63	--	109	--	10
		CV (%)			--	15.2	--	9.3	--	7.8	--	6.2

---

(1) $\overline{M}$ = Average Modulus
(2) $\overline{T}$ = Average Tensile Strength
(3) $\overline{E}_b$ = Average Elongation (@ Break)
(4) s = Standard Deviation
(5) F = Firestone
(6) H = Horizons Research
(7) DB = Dumbbell Specimen
(8) R = Cut Ring Specimen
(9) C = Computer Print-out
(10) B = Book, i.e., calculated from Instron chart
(11) CV = Coefficient of Variation

TABLE XVIII

O-RING COMPOUNDS FOR PHYSICAL TESTING AT PARKER SEAL

Compound R-194-	844	845	846	847
Polymer (K-17638)	100	100	100	100
Quso WR-82	30	30	30	25
FEF Black	--	--	--	5
Stan Mag ELC	6	6	6	6
$(8\text{-HQ})_2Zn$ (Stabilizer)	2	2	2	2
Union Carbide Silane A-151	2	--	--	--
" " " A-1100	--	2	--	--
" " " A-174	--	--	2	--
Teflon 6	--	--	--	5
Silastic 430	--	--	--	5
Vulcup R	0.4	0.4	0.4	0.4

Mixing Procedure -- Polymer and Quso WR82 were mixed in a Brabender -- Dow Corning silanes were added and the mixing continued for 10 minutes -- the Stan Mag ELC was then added and the masterbatch dumped and cooled -- the remaining pigments and curing agent was added to the masterbatch on a rubber mill.

	844		845		846		847	
Rubber Mill Processing	P[1]		P		P		G[2]	
Stress-Strain	R[3]	DB[4]	R	DB	R	DB	R	DB
Press Cure - 30' @ 320°F								
Post Cure - 1 hr. @ 350°F								
50% M (psi)	591	1183	362	818	570	1140	725	1262
100% M (psi)	--	--	1158	--	1383	--	1365	1700
Tensile Strength (psi)	1500	1307	1491	1393	1383	1261	1608	1700
E_b (%)	100	60	123	80	100	55	137	105
% Tension Set (@ Break)	--	3	--	3	--	4	--	6
Shore A Hardness	60		60		75		67	
Press Cure - 40' @ 340°F								
Post Cure - 1 hr. @ 350°F								
Compression Set (%)								
70 hr. @ 300°F	22		32		32		30	
70 hr. @ 350°F	40		56		48		47	
Tensile Strength (73°F)(ppi)[5]	74		61		--		207	

(1) P = Poor
(2) G = Good
(3) Cut Ring Specimen
(4) Dumbbell Specimen
(5) Same cure as Hardness Specimen

TABLE XVIII (CONTD.)

O-RING COMPOUNDS FOR PHYSICAL TESTING AT PARKER SEAL

Compound R-194-	844	845	846	847
Brabender Extrusion				
(2:1 Screw; 40 RPM; 1/8" Diameter Die)				
Barrel (t °C)	203	126	149	203
Head (t °C)	145	158	192	144
Stock (t °C)	190	150	180	200
% Die Swell	6	6	6	6
Extrusion Rate (g/min)	32.4	36.0	28.4	42.6
Surface Appearance	rough	rough	rough	rough

O-Ring Construction

Compound R-194-	844	845	846					847
Mode of Construction(6)	E,O	E,O	SWG	SAG	S	C	E,O	E,O
Weight of Sample (g)	3.4	3.4	4.2	4.5	4.5	4.0	3.5	3.5
Press Cure (22'/ 340°F)								
Post Cure (1 hr./ 350°F)								
50% M (psi)	257	213	424	388	478	433	390	262
100% M (psi)	--	971	--	--	--	--	--	667
Tensile Strength (psi)	1053	1159	1030	1046	1104	949	1105	1200
E_b (%)	99	111	84	90	87	82	94	153
Failure								
Appearance of O-Ring	E(7)	E	E	E	E	E	E	E

Compound R-194-	844	845	846	847
Stress-Strain (Cut-Ring)				
Press Cure - 40' @ 340°F				
Post Cure - 1 hr. @ 350°F				
50% M (psi)(Original)	342	385	457	427
Aged 240 hr. @ 275°F (Air)	381	480	514	497
300°F "	411	482	494	411
350°F "	347	356	380	456
400°F "	--	--	--	158
73°F (H_3PO_4)	--	247	410	259
" (HCl)	--	198	238	243
" (HNO_3)	--	78	118	89
" (H_2SO_4)	--	00	00	118

(6) E = Extruded Tube; O = Overlap Ends @ 45° Cut; SWG - cut strip with mill grain; SAG - cut strip against mill grain; S = cut out small square slab; C = die-out O-ring shape

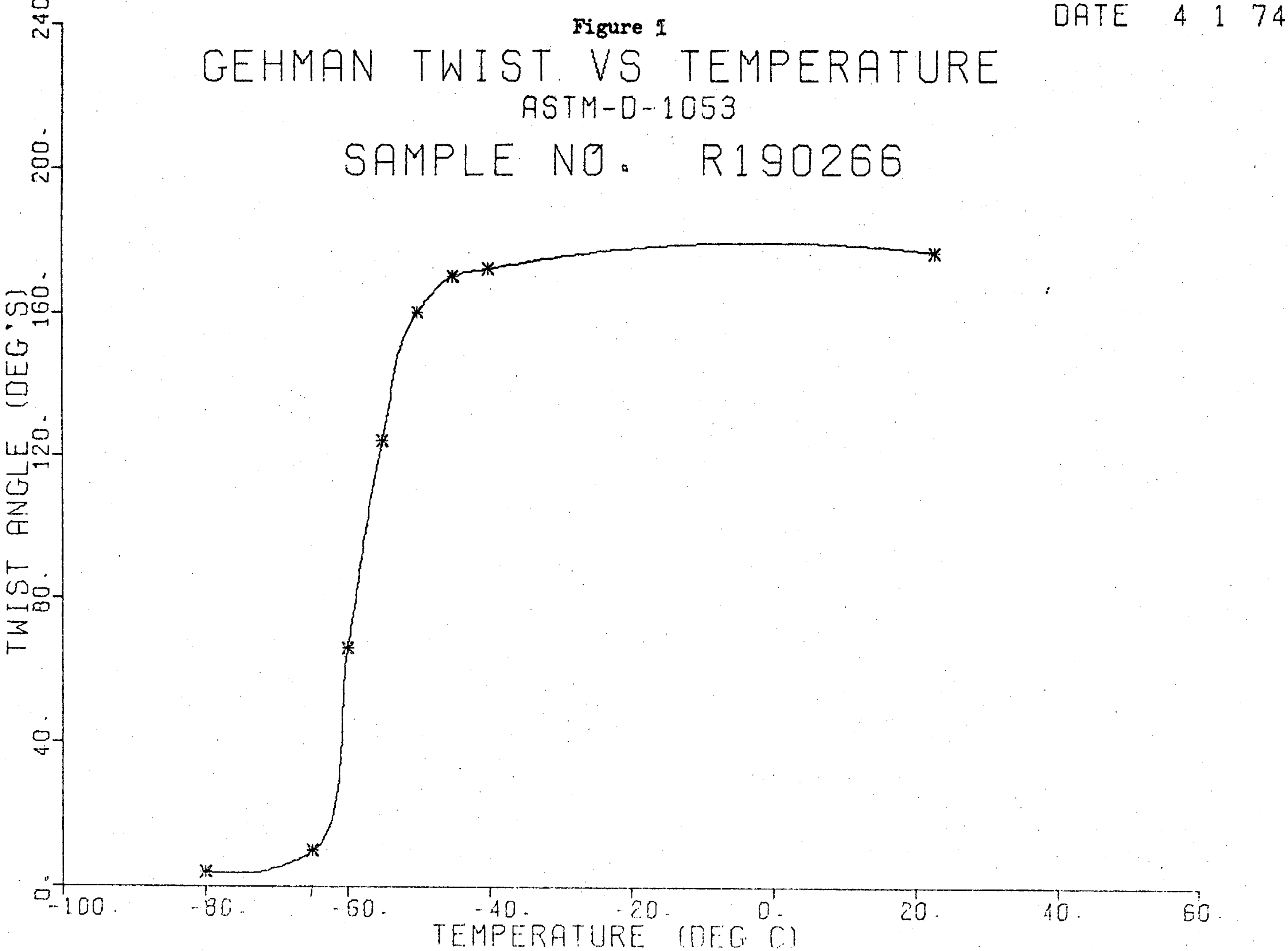
(7) E = Excellent

TABLE XVIII (CONTD.)

O-RING COMPOUNDS FOR PHYSICAL TESTING AT PARKER SEAL

Compound R-194-		844	845	846	847
Stress-Strain (Cut-Ring)(contd.)					
Press Cure - 40' @ 340°F					
Post Cure - 1 hr. @ 350°F					
Tensile Strength (psi)(Original)		988	1472	1187	1081
Aged 240 hr. @ 275°F	(Air)	891	1447	1199	1160
300°F	"	1108	1173	1028	914
350°F	"	820	907	727	809
400°F	"	177	155	160	152
73°F	(H_3PO_4)	--	1089	946	948
"	(HCl)	--	355	272	373
"	(HNO_3)	--	182	114	225
"	(H_2SO_4)	--	90	57	176
E_b (%) (Original)		95	100	95	125
Aged 240 hr. @ 275°F	(Air)	90	103	100	125
300°F	"	100	96	90	120
350°F	"	105	110	100	125
400°F	"	30	17	20	63
73°F	(H_3PO_4)	--	110	95	135
"	(HCl)	--	70	65	90
"	(HNO_3)	--	80	55	120
"	(H_2SO_4)	--	40	30	100
ASTM Fuel A					
% Wt. Change		1.1	1.0	1.0	3.0
% Vol. Swell		3.4	3.3	3.2	9.0
% Extracted		0.4	0.6	0.4	0.5
ASTM Fuel B					
% Wt. Change		2.4	2.3	2.0	4.1
% Vol. Swell		7.8	7.2	7.7	14.6
% Extracted		1.7	1.6	1.5	2.2
ASTM Fuel C					
% Wt. Change		2.9	3.1	2.8	5.7
% Vol. Swell		9.0	10.0	9.6	15.9
% Extracted					
Mil-H-5606-C					
% Wt. Change		0.1	0.1	0.1	0.5
% Vol. Swell		0.4	-0.3	0.9	1.8
% Extracted		0.4	0.4	0.5	0.3

Figure 1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GEHMAN TWIST VS TEMPERATURE
ASTM-D-1053
SAMPLE NØ. R190266
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40.
80.
120.
160.
200.
240
TEMPERATURE (DEG C)
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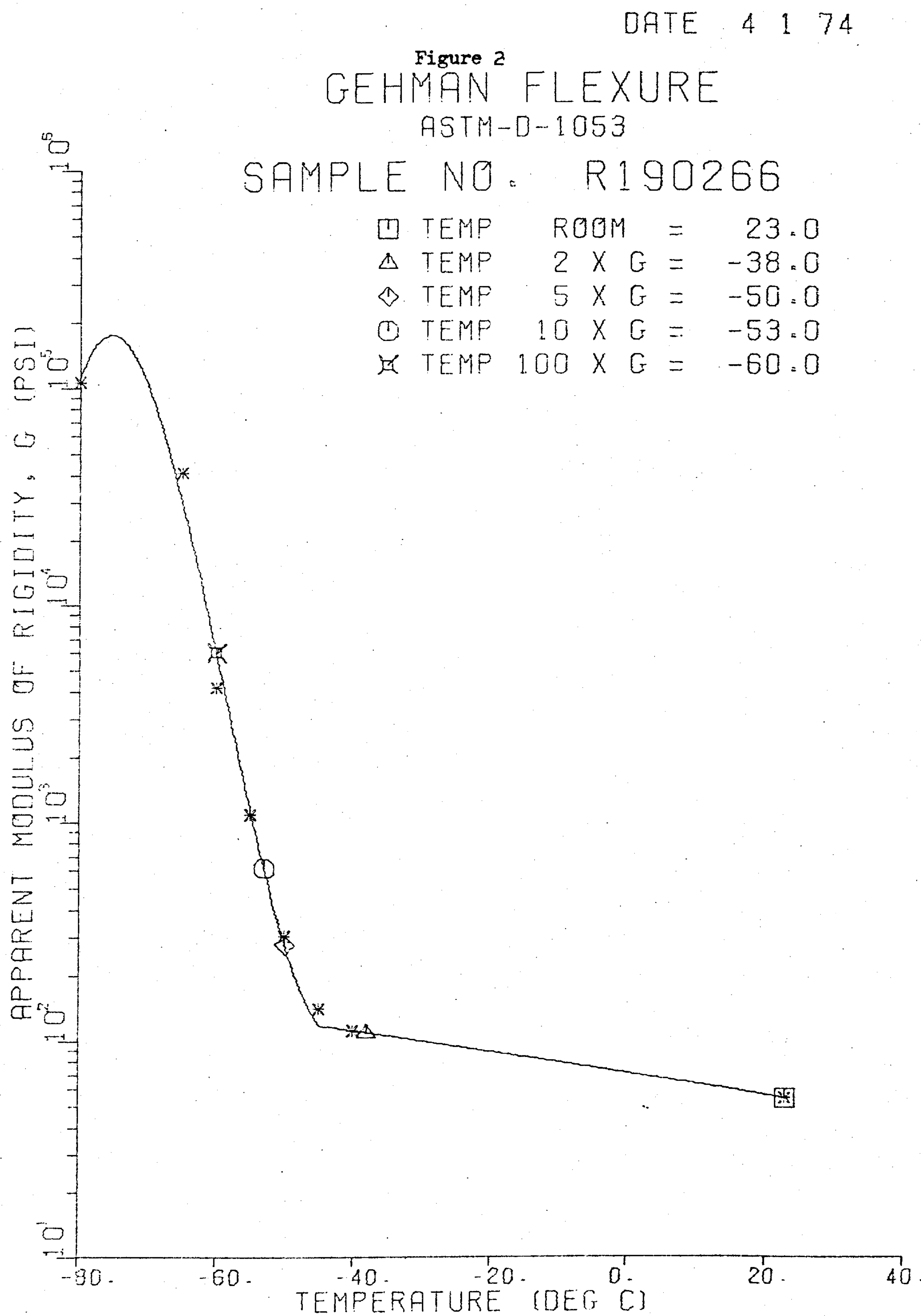
-80.
-60.
-40.
-20.
0.
20.
40.
60.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GEHMAN FLEXURE
ASTM-D-1053
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TEMP 5 X G = -50.0
TEMP 10 X G = -53.0
TEMP 100 X G = -60.0
APPARENT MODULUS OF RIGIDITY, G (PSI)
TEMPERATURE (DEG C)
-80.
-60.
-40.
-20.
0.
20.
40.

Figure 2

GEHMAN FLEXURE
ASTM D-1053
YELLOW CODE WIRE

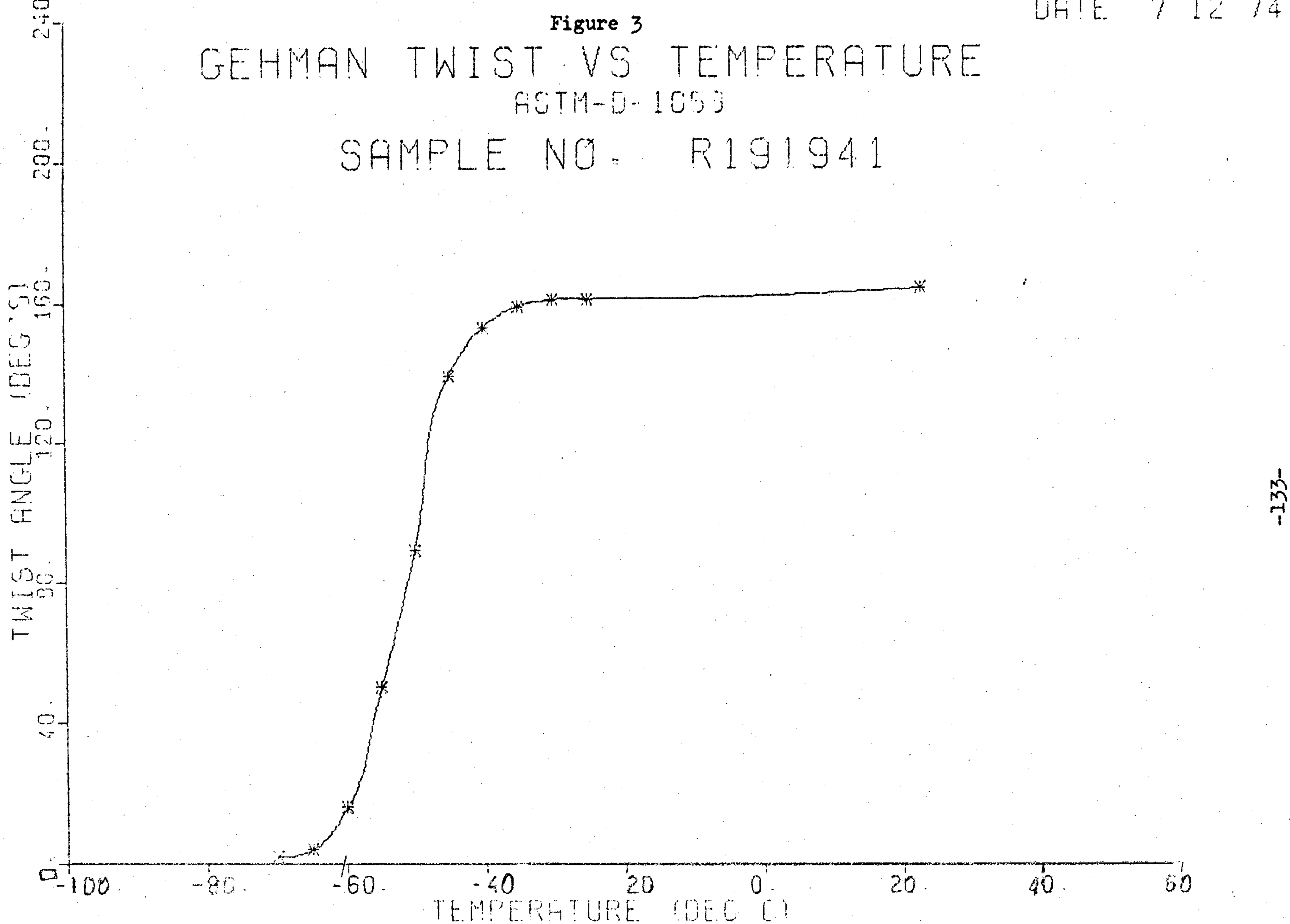
SAMPLE NO. R190266
DATE 4 1 74

A = 0.1250
B = 0.0730
MU = 3.3960
K = 0.5000

TEMP DEG C	X DEG,	G PSI
23.0	176.0	54.7
-40.0	172.0	111.9
-45.0	170.0	141.5
-50.0	160.0	300.8
-55.0	124.0	1087.0
-60.0	66.0	4157.6
-65.0	10.0	40920.3
-80.0	4.0	105911.6

ROOM TEMPERATURE	23.0	GEHMAN FLEXURE	54.7 PSI
2 TIMES G	-38.0	GEHMAN FLEXURE	109.4 PSI
5 TIMES G	-50.0	GEHMAN FLEXURE	278.4 PSI
10 TIMES G	-53.0	GEHMAN FLEXURE	619.5 PSI
100 TIMES G	-60.0	GEHMAN FLEXURE	6045.9 PSI

Figure 3


DATE 7 12 74
GEHMAN TWIST VS TEMPERATURE
ASTM-D-1053
SAMPLE NO. R191941
TWIST ANGLE (DEG'S)
0
40
80
120
160
200
240
-100
-80
-60
-40
-20
0
20
40
60
TEMPERATURE (DEG C)

DATE 7 12 74

Figure 4

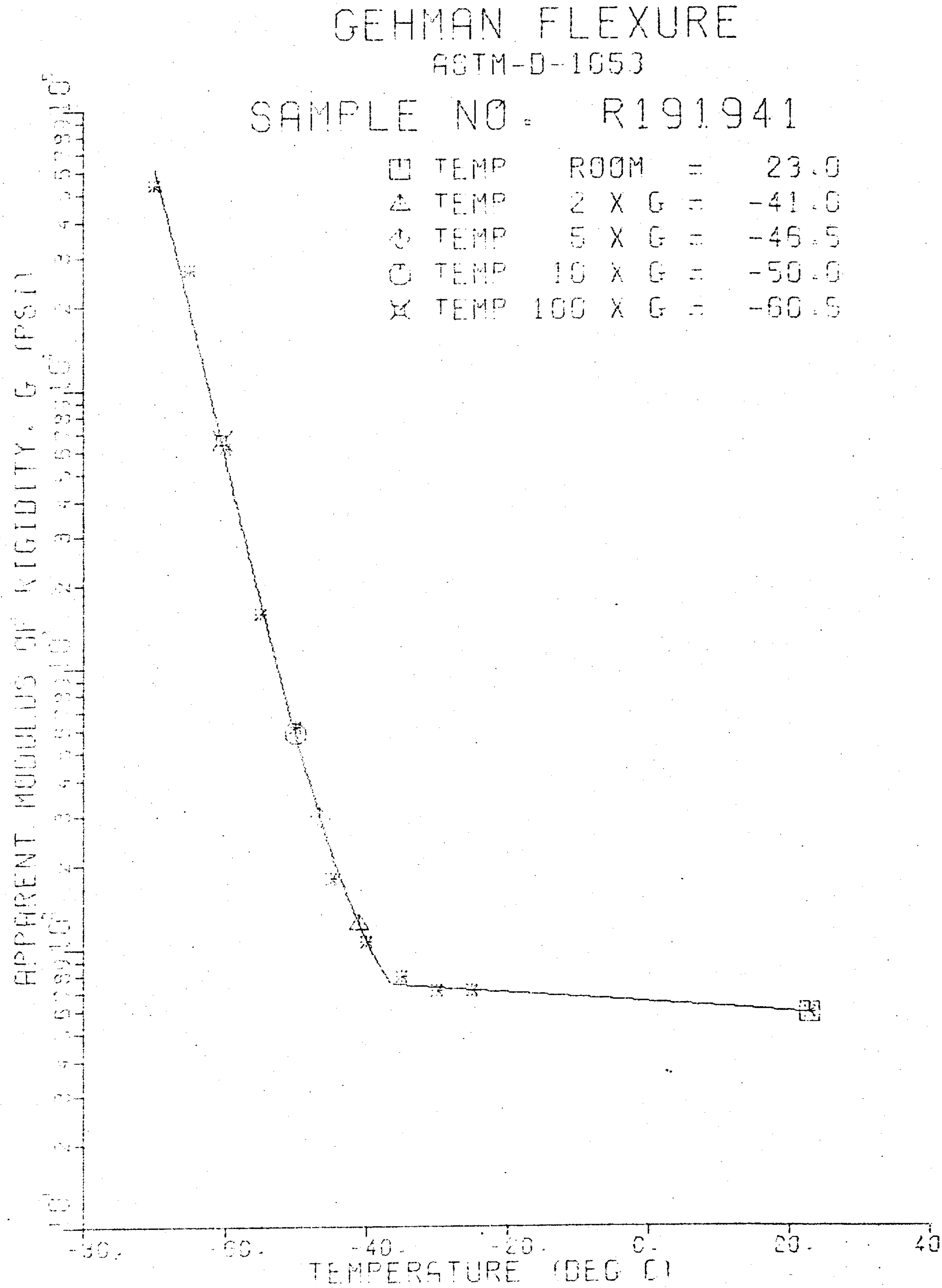


TABLE XX
GEHMAN FLEXURE
ASTM D-1053
BLACK CODE WIRE

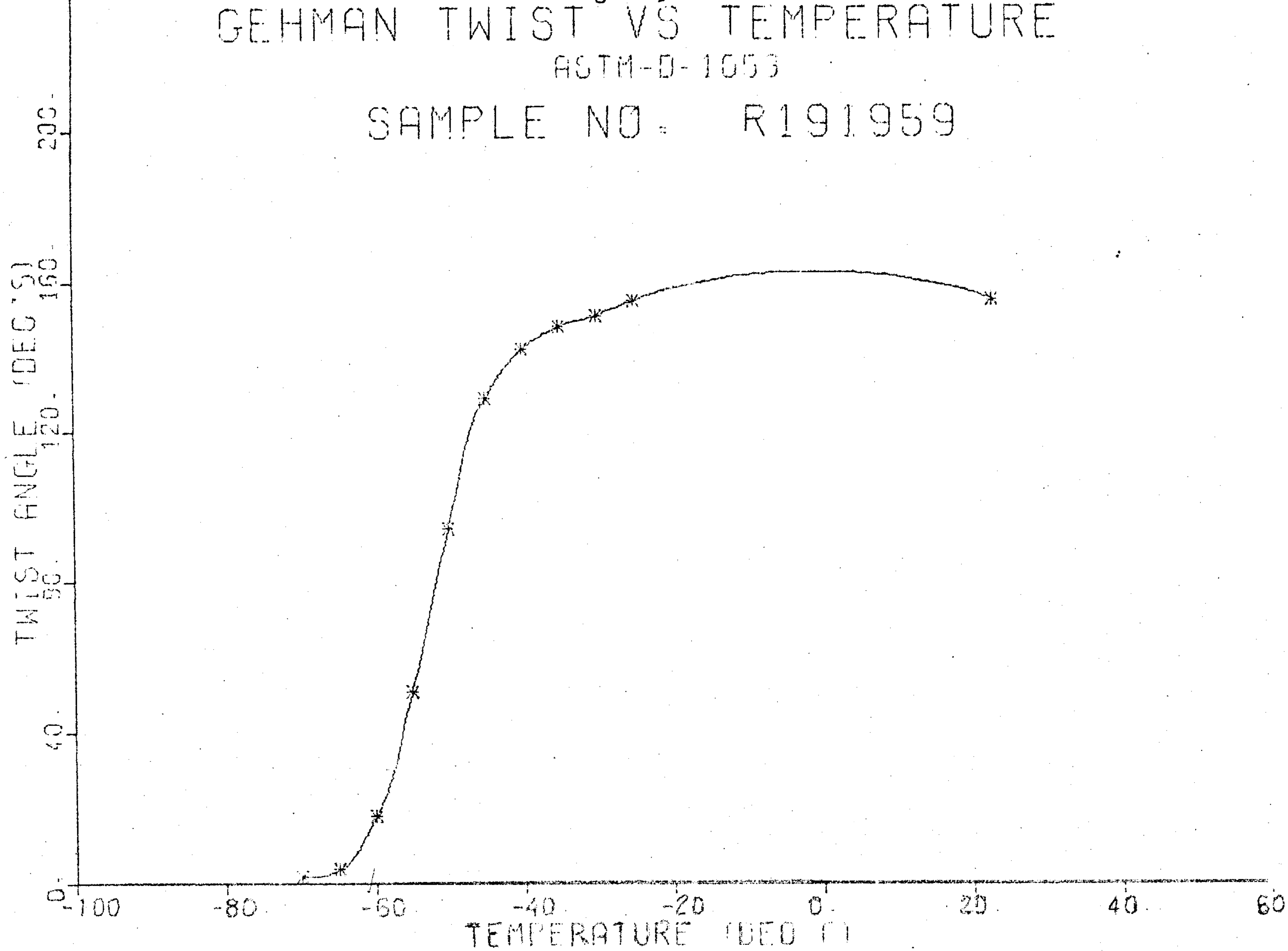
SAMPLE NO. R191941
DATE 7 12 74

A = 0.1250
B = 0.0730
MU = 3.3960
K = 0.1250

TEMP DEG C	X DEG,	G PSI
23.0	164.0	58.7
-25.0	161.0	71.0
-30.0	161.0	71.0
-35.0	159.0	79.4
-40.0	153.0	106.1
-45.0	139.0	177.5
-50.0	89.0	615.2
-55.0	50.0	1564.6
-60.0	16.0	6168.1
-65.0	4.0	26477.9
-70.0	2.0	53557.5

ROOM TEMPERATURE	23.0	GEHMAN FLEXURE	58.7 PSI
2 TIMES G	-41.0	GEHMAN FLEXURE	122.5 PSI
5 TIMES G	-46.5	GEHMAN FLEXURE	295.0 PSI
10 TIMES G	-50.0	GEHMAN FLEXURE	587.4 PSI
100 TIMES G	-60.5	GEHMAN FLEXURE	6621.2 PSI

Figure 5


DATE 7 12 74
GEHMAN TWIST VS TEMPERATURE
ASTM-D-1053
SAMPLE NO. R191959
TWIST ANGLE (DEG'S)
0
40
80
120
160
200
240
TEMPERATURE (DEG C)
-100
-80
-60
-40
-20
0
20
40
60

DATE 7 12 74

Figure 6

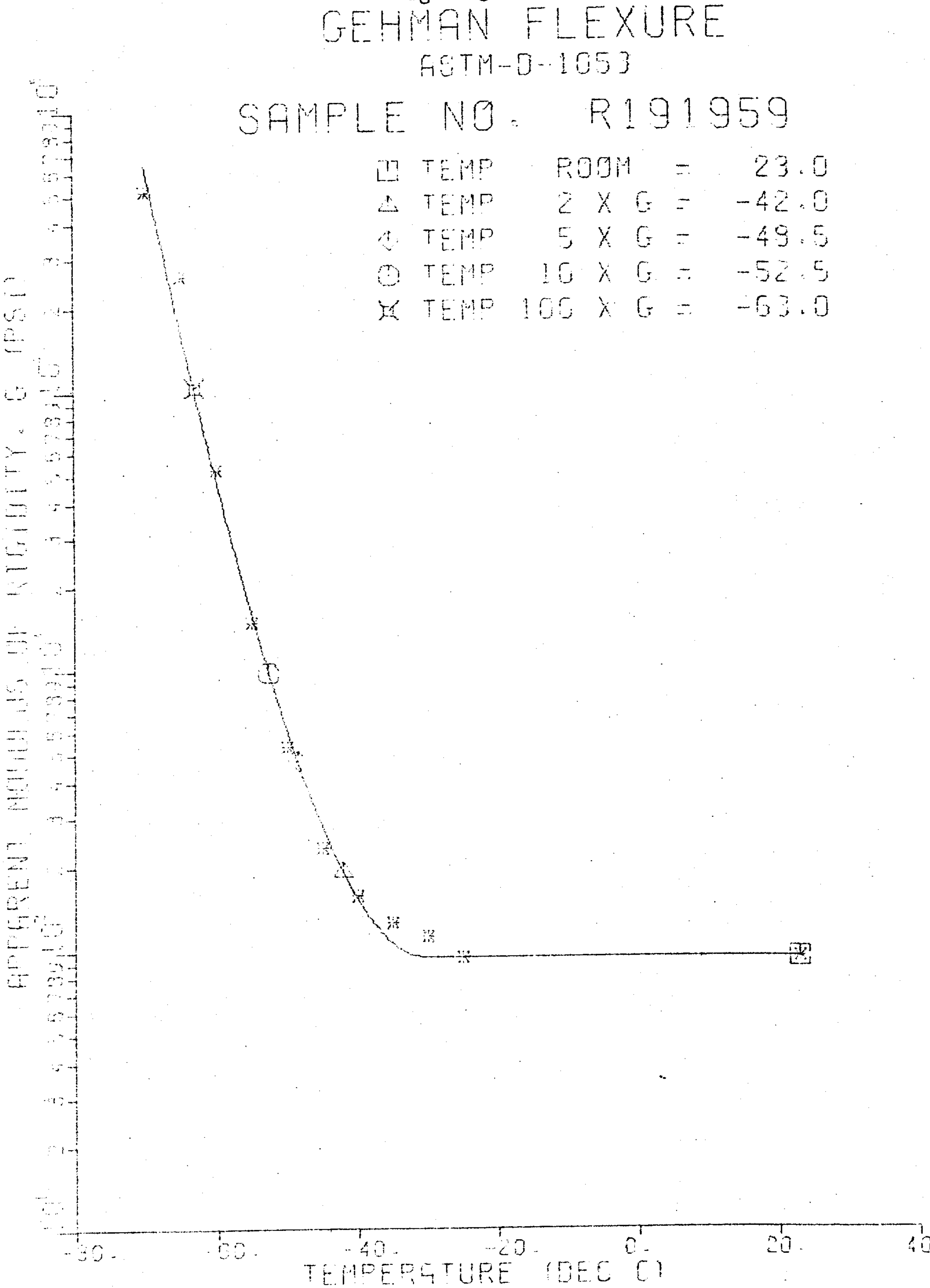


TABLE XXI
GEHMAN FLEXURE
ASTM D-1053
BLACK CODE WIRE

SAMPLE NO. R191959
DATE 7 12 74

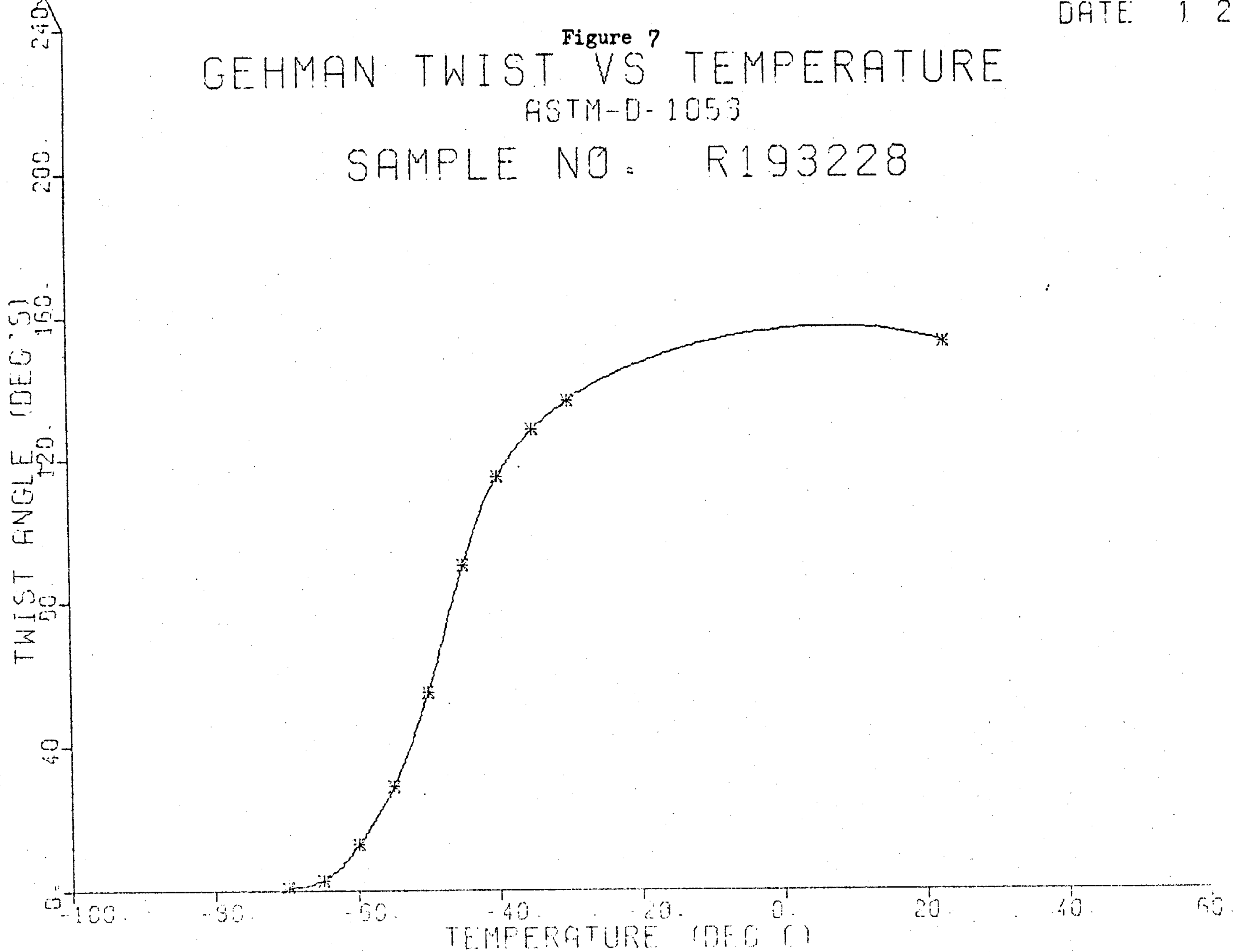
A = 0.1250
B = 0.0740
MU = 3.3640
K = 0.1250

TEMP DEG C	X DEG,	G PSI
23.0	155.0	94.0
-25.0	155.0	94.0
-30.0	151.0	112.0
-35.0	148.0	126.0
-40.0	142.0	156.0
-45.0	129.0	230.5
-50.0	94.0	533.5
-55.0	51.0	1475.1
-60.0	18.0	5248.7
-65.0	4.0	25660.7
-70.0	2.0	51904.6

ROOM TEMPERATURE	23.0	GEHMAN FLEXURE	94.0 PSI
2 TIMES G	-42.0	GEHMAN FLEXURE	189.4 PSI
5 TIMES G	-48.5	GEHMAN FLEXURE	473.9 PSI
10 TIMES G	-52.5	GEHMAN FLEXURE	974.9 PSI
100 TIMES G	-63.0	GEHMAN FLEXURE	10312.0 PSI

DATE 1 29 75

Figure 7



DATE 1 29 75

Figure 8

GEHMAN FLEXURE

ASTM-D-1053

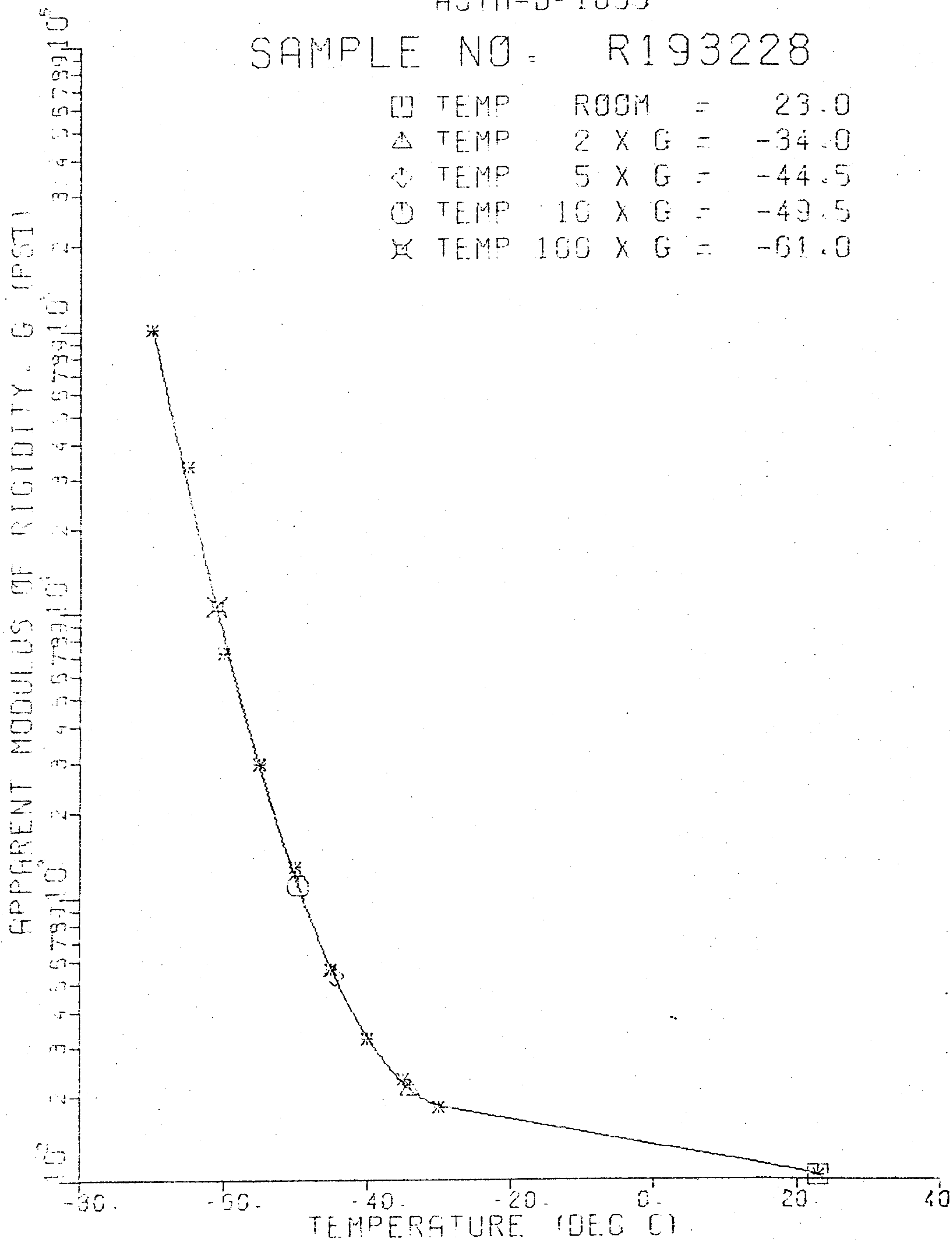


TABLE XXII
GEHMAN FLEXURE
ASTM D-1053
BLACK CODE WIRE

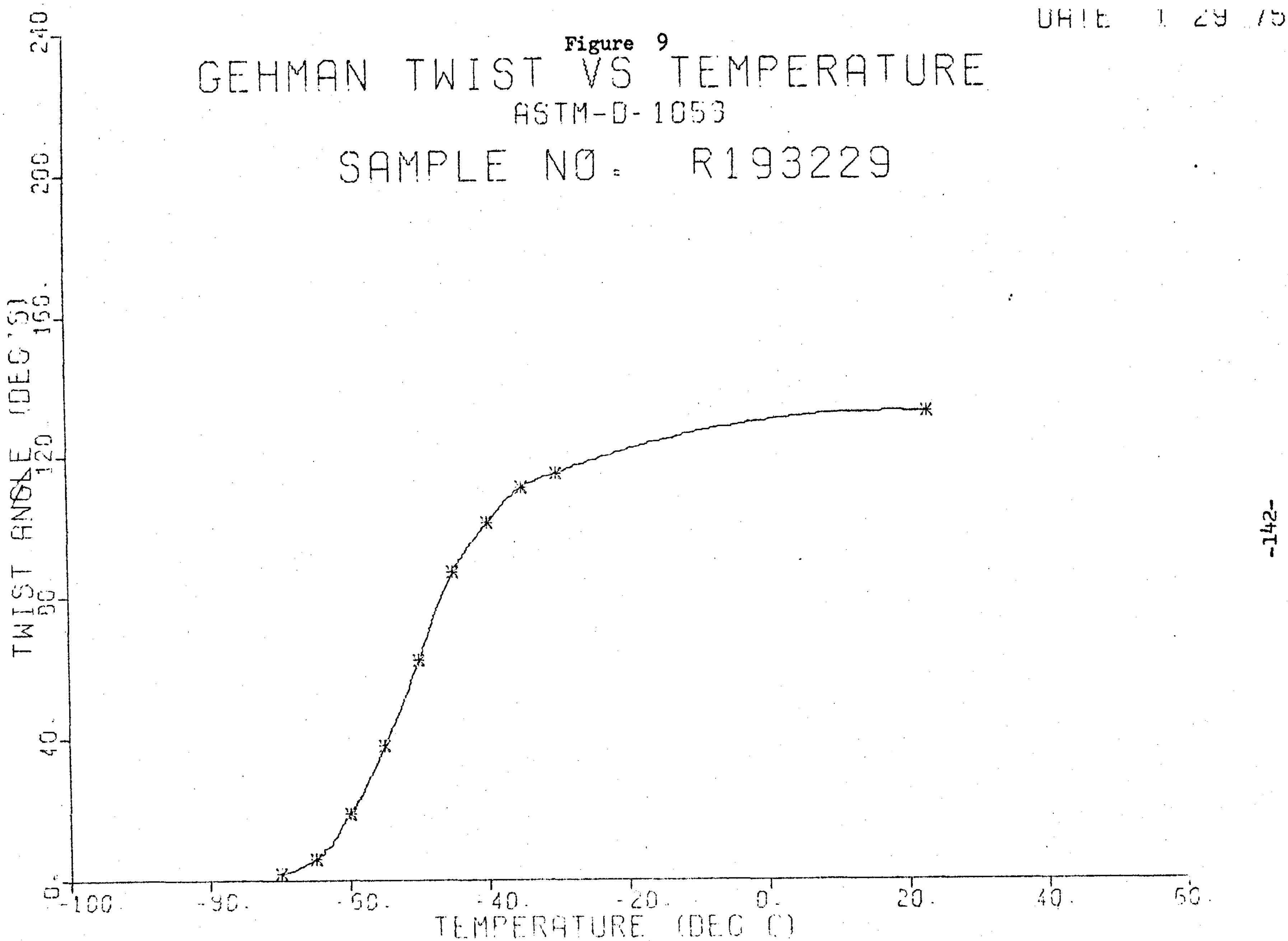
SAMPLE NO. R193228
DATE 1 29 75

A = 0.1250
B = 0.0750
MU = 3.3410
K = 0.1250

TEMP DEG C	X DEG,	G PSI
23.0	152.0	103.9
-30.0	136.0	182.4
-35.0	128.0	229.1
-40.0	115.0	318.8
-45.0	90.0	564.0
-50.0	55.0	1281.9
-55.0	29.0	2936.8
-60.0	13.0	7245.6
-65.0	3.0	33278.1
-70.0	1.0	100962.5

ROOM TEMPERATURE	23.0	GEHMAN FLEXURE	103.9 PSI
2 TIMES G	-34.0	GEHMAN FLEXURE	211.2 PSI
5 TIMES G	-44.5	GEHMAN FLEXURE	537.8 PSI
10 TIMES G	-49.5	GEHMAN FLEXURE	1105.0 PSI
100 TIMES G	-61.0	GEHMAN FLEXURE	10639.7 PSI

Figure 9


DATE 1 29 75
GEHMAN TWIST VS TEMPERATURE
ASTM-D-1053
SAMPLE NO. R193229
TWIST ANGLE (DEG'S)
0.
40.
80.
120.
160.
200.
240.
TEMPERATURE (DEG C)
-100.
-80.
-60.
-40.
-20.
0.
20.
40.
60.

DATE 1 29 75

Figure 10

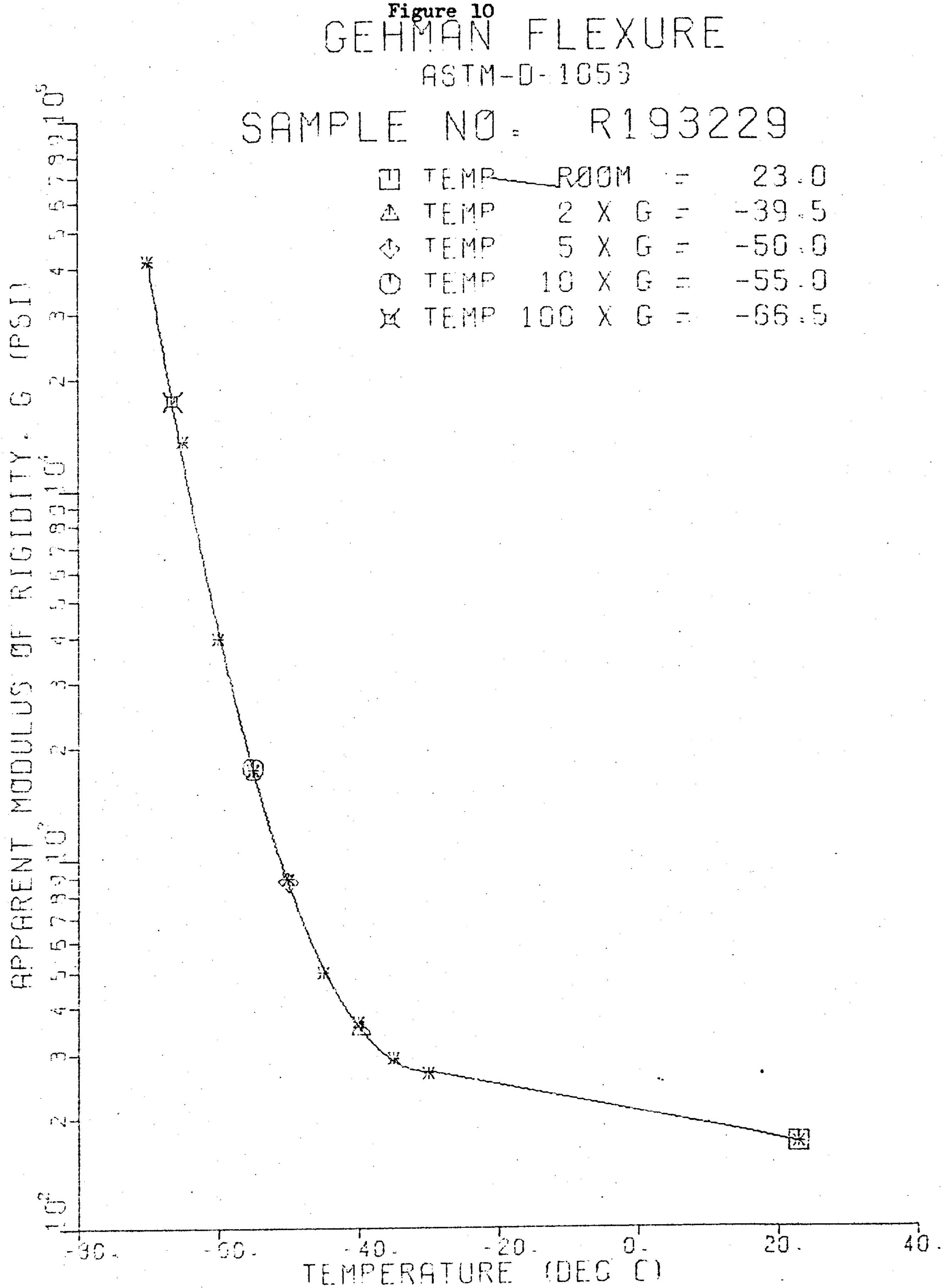


TABLE XXIII

GEHMAN FLEXURE

ASTM D-1053

BLACK CODE WIRE

SAMPLE NO. R193229

DATE 1 29 75

A = 0.1250
B = 0.0810
MU = 3.1960
K = 0.1250

TEMP DEG C	X DEG,	G PSI
23.0	132.0	170.2
-30.0	115.0	264.5
-35.0	111.0	290.9
-40.0	101.0	366.1
-45.0	87.0	500.3
-50.0	62.0	890.8
-55.0	38.0	1749.0
-60.0	19.0	3966.2
-65.0	6.0	13573.8
-70.0	2.0	41657.7

ROOM TEMPERATURE	23.0	GEHMAN FLEXURE	170.2 PSI
2 TIMES G	-39.5	GEHMAN FLEXURE	346.8 PSI
5 TIMES G	-50.0	GEHMAN FLEXURE	875.8 PSI
10 TIMES G	-55.0	GEHMAN FLEXURE	1762.2 PSI
100 TIMES G	-66.5	GEHMAN FLEXURE	17550.7 PSI

Figure 11

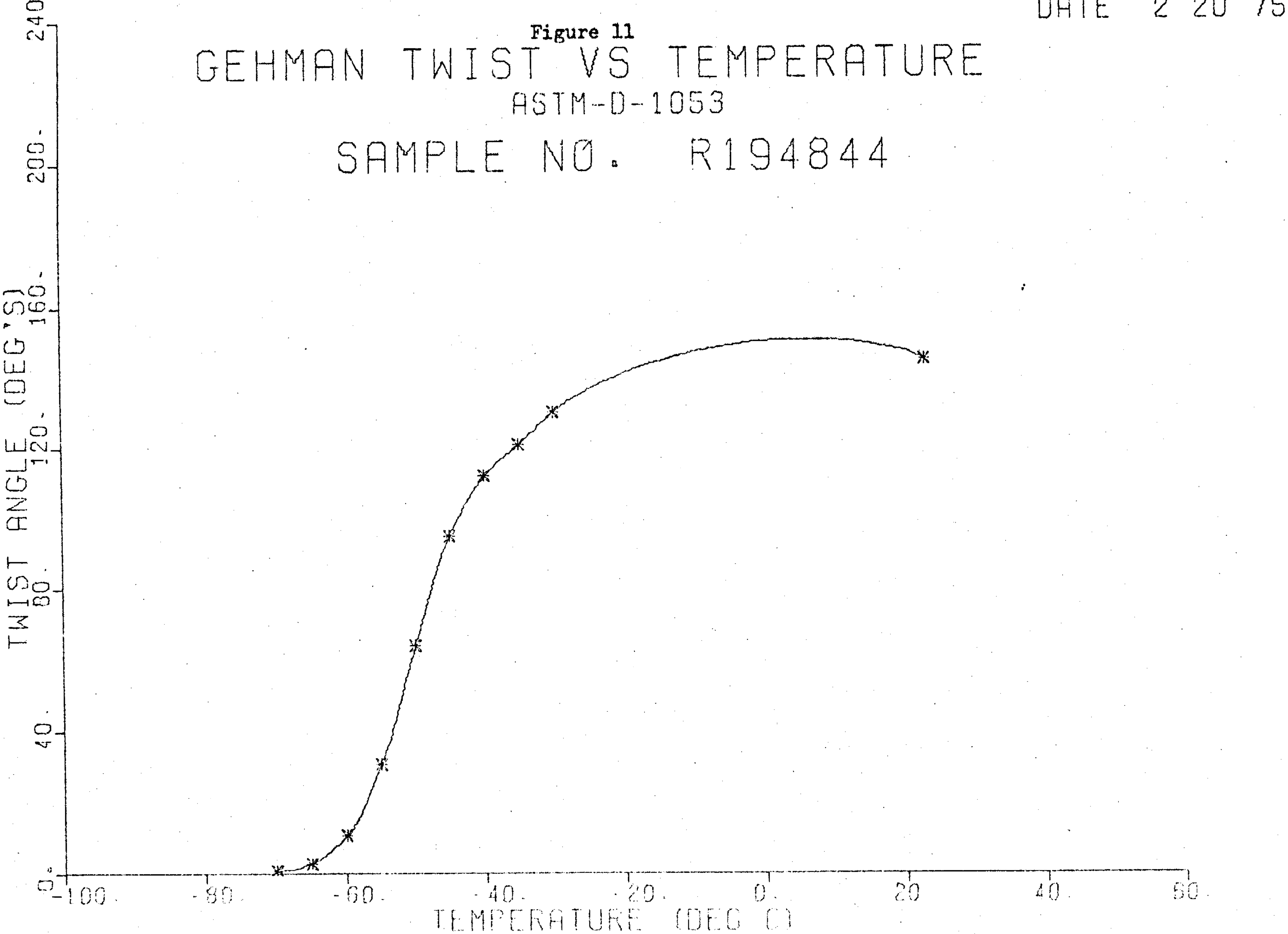

DATE 2 20 75
GEHMAN TWIST VS TEMPERATURE
ASTM-D-1053
SAMPLE NO. R194844
TWIST ANGLE (DEG'S)
0.
40.
80.
120.
160.
200.
240
-100
-80.
-60.
-40.
-20.
0.
20
40
60
TEMPERATURE (DEG C)

Figure 12

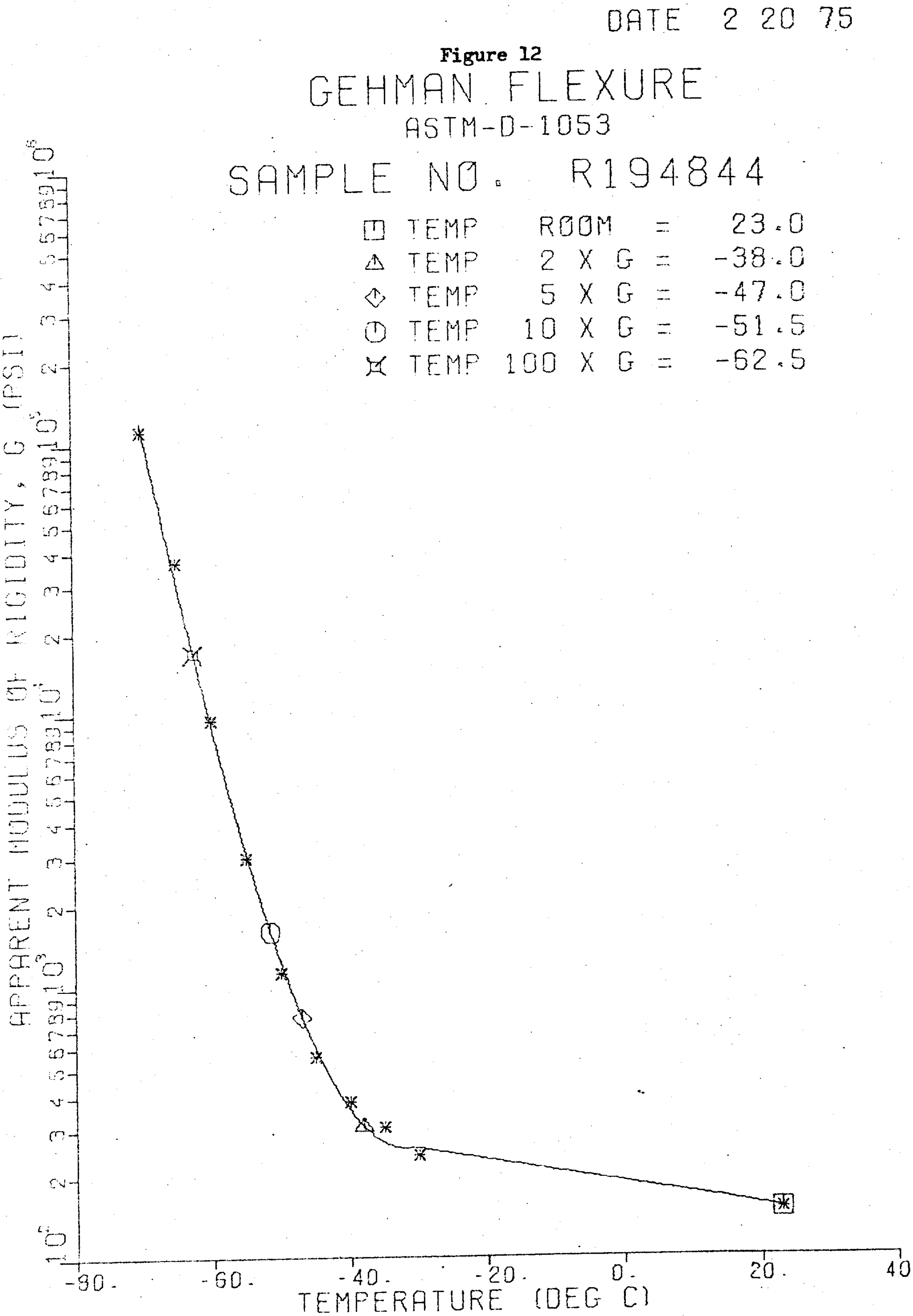

DATE 2 20 75
GEHMAN FLEXURE
ASTM-D-1053
SAMPLE NØ. R194844
TEMP ROØM = 23.0
TEMP 2 X G = -38.0
TEMP 5 X G = -47.0
TEMP 10 X G = -51.5
TEMP 100 X G = -62.5
APPARENT MODULUS OF RIGIDITY, G (PSI)
TEMPERATURE (DEG C)
-80.
-60.
-40.
-20.
0.
20.
40

TABLE XXIV
GEHMAN FLEXURE
ASTM D-1053
BLACK CODE WIRE

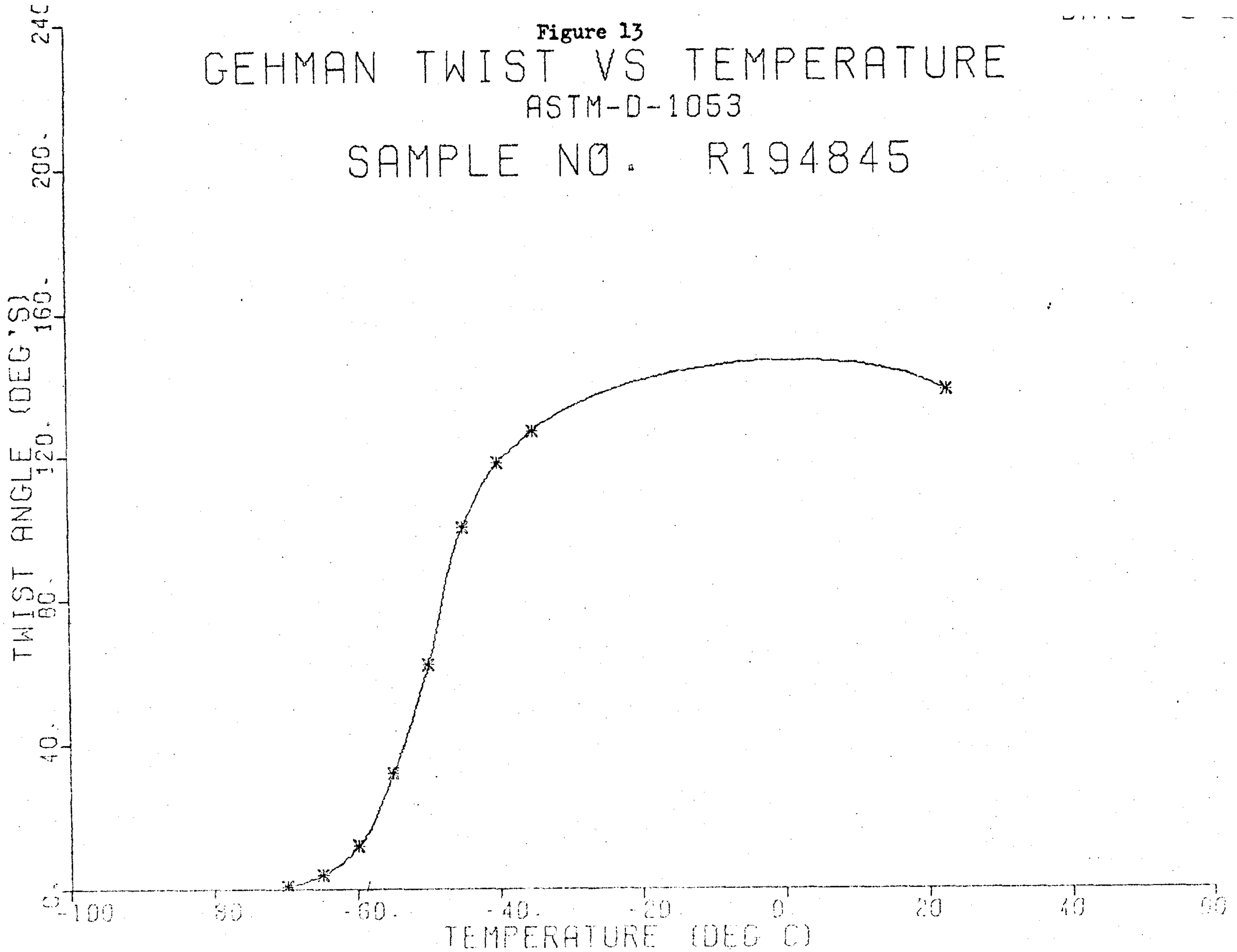
SAMPLE NO. R194844
DATE 2 20 75

A = 0.1250
B = 0.0720
MU = 3.4170
K = 0.1250

TEMP DEG C	X DEG.	G PSI
23.0	145.0	150.4
-30.0	130.0	239.7
-35.0	121.0	303.9
-40.0	112.0	378.4
-45.0	95.0	557.7
-50.0	64.0	1129.8
-55.0	31.0	2996.0
-60.0	11.0	9576.7
-65.0	3.0	36777.0
-70.0	1.0	111577.7

ROOM TEMPERATURE	23.0	GEHMAN FLEXURE	150.4 PSI
2 TIMES G	-38.0	GEHMAN FLEXURE	307.2 PSI
5 TIMES G	-47.0	GEHMAN FLEXURE	780.3 PSI
10 TIMES G	-51.5	GEHMAN FLEXURE	1606.6 PSI
100 TIMES G	-62.5	GEHMAN FLEXURE	16864.0 PSI

Figure 13


GEHMAN TWIST VS TEMPERATURE
ASTM-D-1053
SAMPLE NO. R194845
TWIST ANGLE (DEG'S)
TEMPERATURE (DEG C)
-100
-80
-60
-40
-20
0
20
40
60
0
40
80
120
160
200
240

DATE 2 20 75

Figure 14

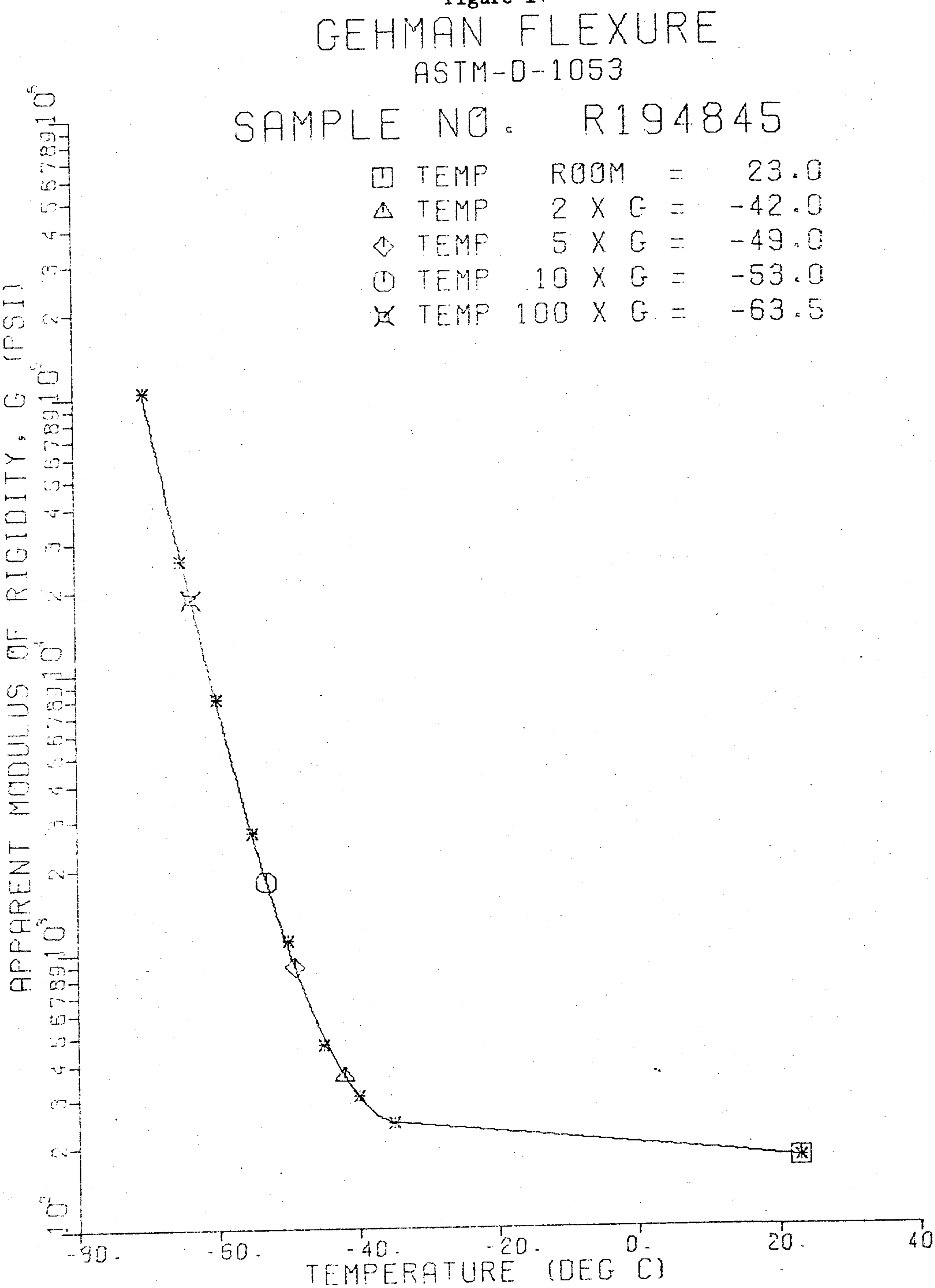


TABLE XXV
GEHMAN FLEXURE
ASTM D-1053
BLACK CODE WIRE

SAMPLE NO. R194845
DATE 2 20 75

A = 0.1250
B = 0.0740
MU = 3.3640
K = 0.1250

TEMP DEG C	X DEG,	G PSI
23.0	138.0	177.4
-35.0	127.0	243.3
-40.0	118.0	306.4
-45.0	100.0	466.5
-50.0	62.0	1109.9
-55.0	32.0	2697.2
-60.0	12.0	8164.7
-65.0	4.0	25660.7
-70.0	1.0	104392.4

RCCM TEMPERATURE	23.0	GEHMAN FLEXURE	177.4 PSI
2 TIMES G	-42.0	GEHMAN FLEXURE	359.6 PSI
5 TIMES G	-49.0	GEHMAN FLEXURE	887.8 PSI
10 TIMES G	-53.0	GEHMAN FLEXURE	1796.2 PSI
100 TIMES G	-63.5	GEHMAN FLEXURE	18680.9 PSI

Figure 15

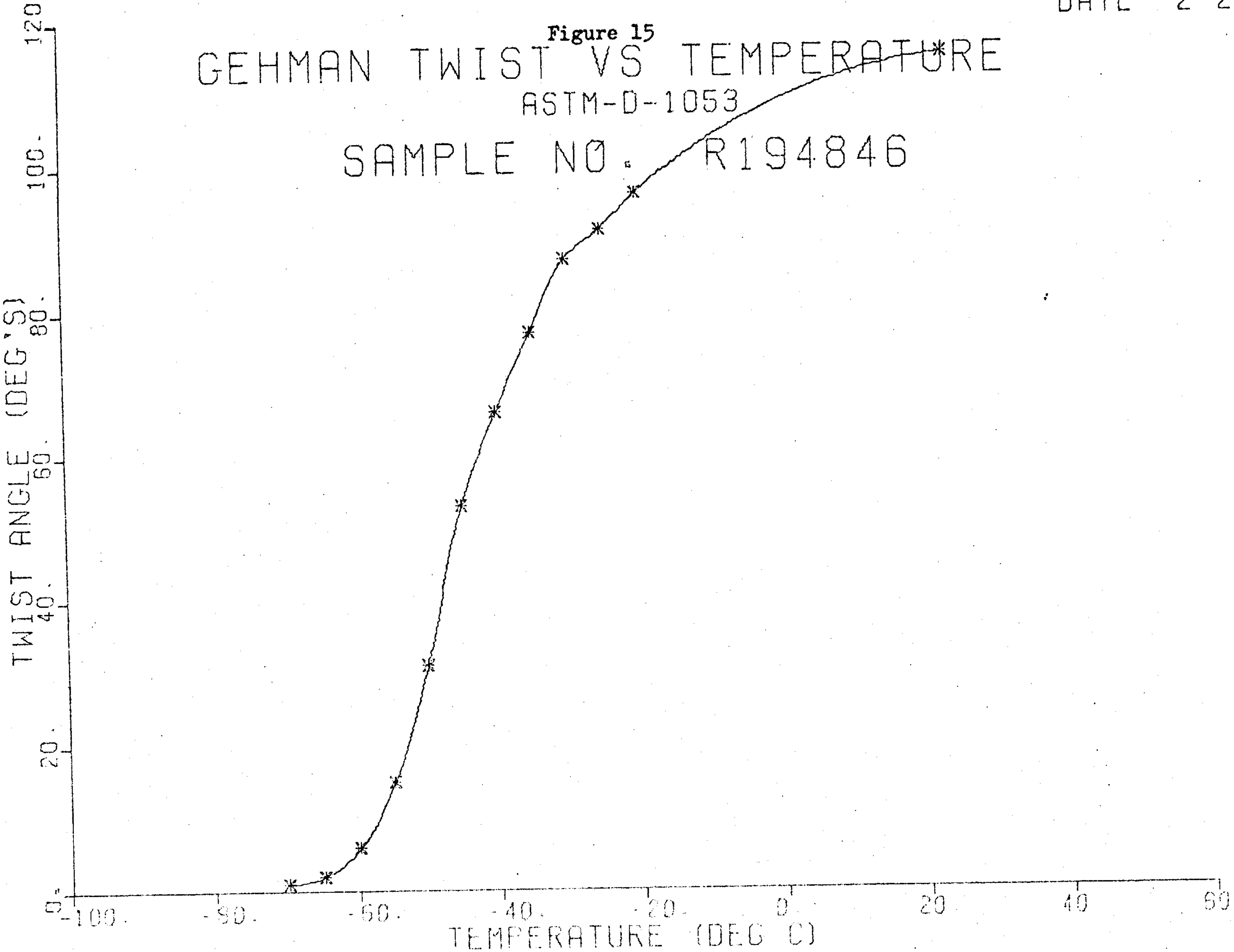

GEHMAN TWIST VS TEMPERATURE
ASTM-D-1053
SAMPLE NO. R194846
TWIST ANGLE (DEG'S)
TEMPERATURE (DEG C)
0.
20.
40.
60.
80.
100.
120.
-100.
-80.
-60.
-40.
-20.
0.
20.
40.
60.

Figure 16

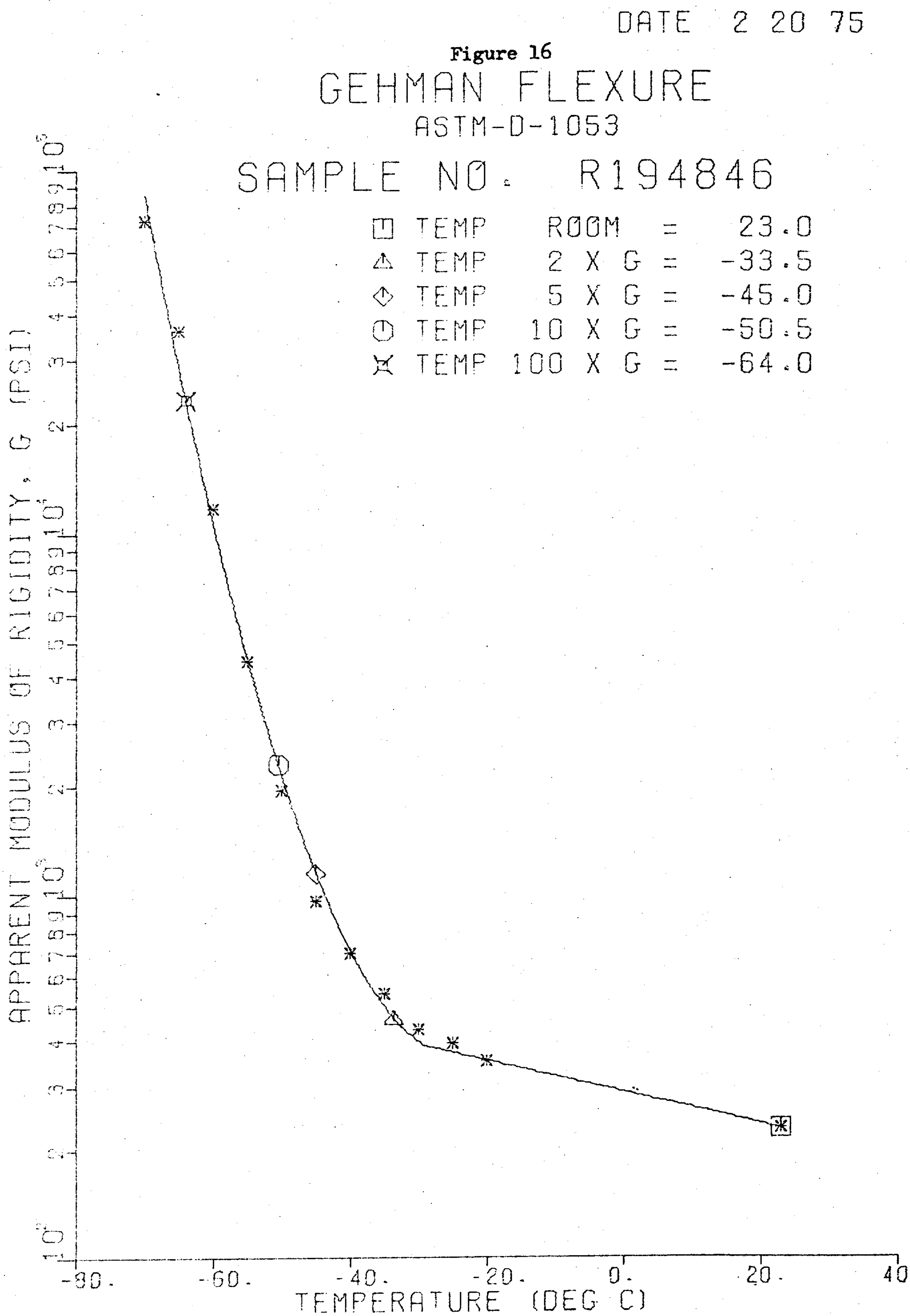

DATE 2 20 75
GEHMAN FLEXURE
ASTM-D-1053
SAMPLE NO. R194846
TEMP ROOM = 23.0
TEMP 2 X G = -33.5
TEMP 5 X G = -45.0
TEMP 10 X G = -50.5
TEMP 100 X G = -64.0
APPARENT MODULUS OF RIGIDITY, G (PSI)
TEMPERATURE (DEG C)
-80.
-60.
-40.
-20.
0.
20.
40

TABLE XXVI

GEHMAN FLEXURE

ASTM D-1053

BLACK CODE WIRE

SAMPLE NO. R194846

DATE 2 20 75

A = 0.1250

B = 0.0840

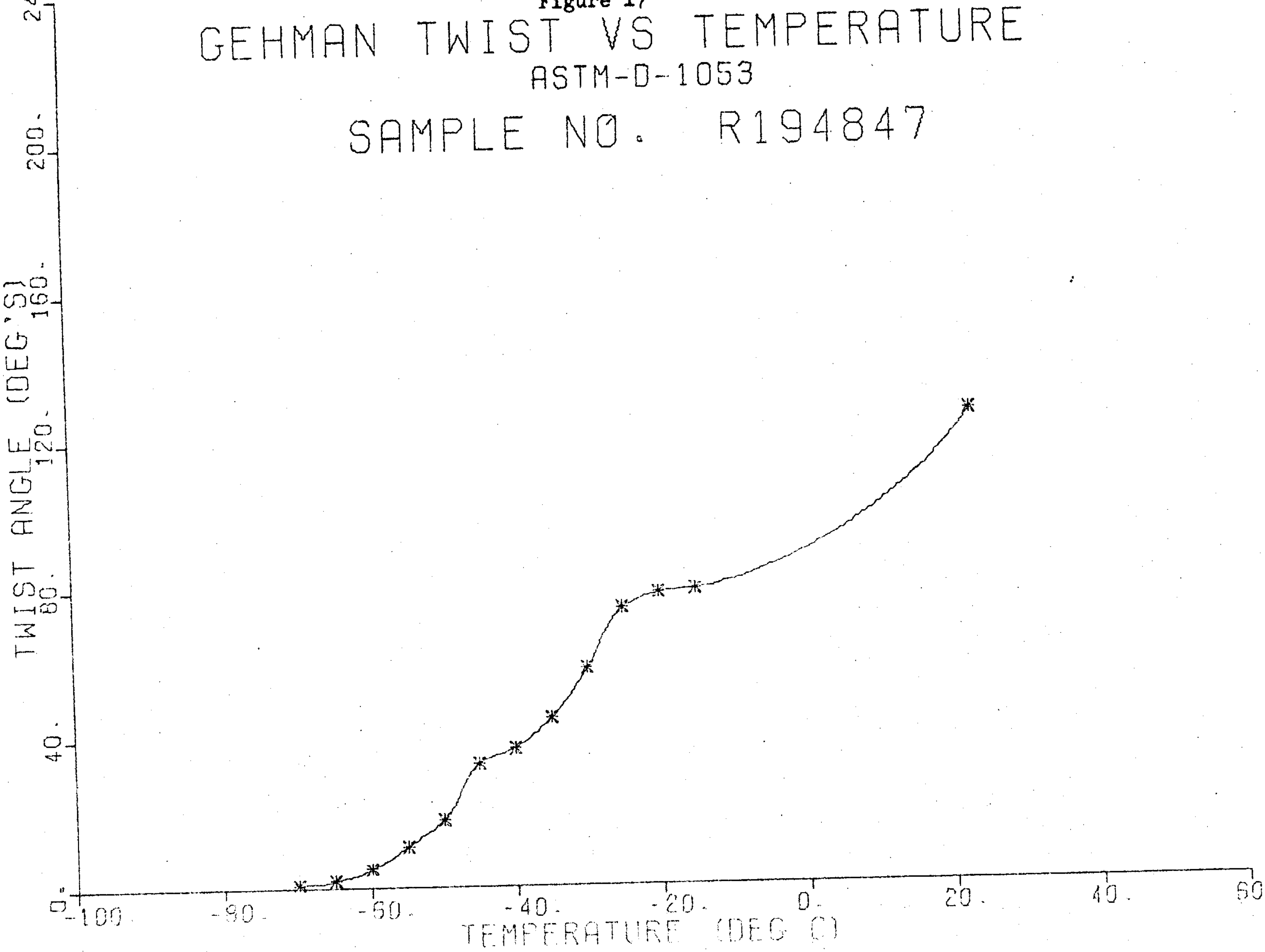
MU = 3.3180

K = 0.1250

TEMP DEG C	X DEG,	G PSI
23.0	115.0	228.4
-20.0	96.0	353.7
-25.0	91.0	395.3
-30.0	87.0	432.1
-35.0	77.0	540.7
-40.0	66.0	698.2
-45.0	53.0	968.6
-50.0	31.0	1943.0
-55.0	15.0	4446.7
-60.0	6.0	11723.3
-65.0	2.0	35978.5
-70.0	1.0	72361.2

ROOM TEMPERATURE	23.0	GEHMAN FLEXURE	228.4 PSI
2 TIMES G	-33.5	GEHMAN FLEXURE	457.9 PSI
5 TIMES G	-45.0	GEHMAN FLEXURE	1155.2 PSI
10 TIMES G	-50.5	GEHMAN FLEXURE	2293.7 PSI
100 TIMES G	-64.0	GEHMAN FLEXURE	23256.0 PSI

Figure 17



GEHMAN TWIST VS TEMPERATURE
ASTM-D-1053
SAMPLE NO. R194847
TWIST ANGLE (DEG'S)
0.
40.
80.
120.
160.
200.
240
-100
-80.
-60.
-40.
-20.
0.
20.
40.
60.
TEMPERATURE (DEG C)

Figure 18

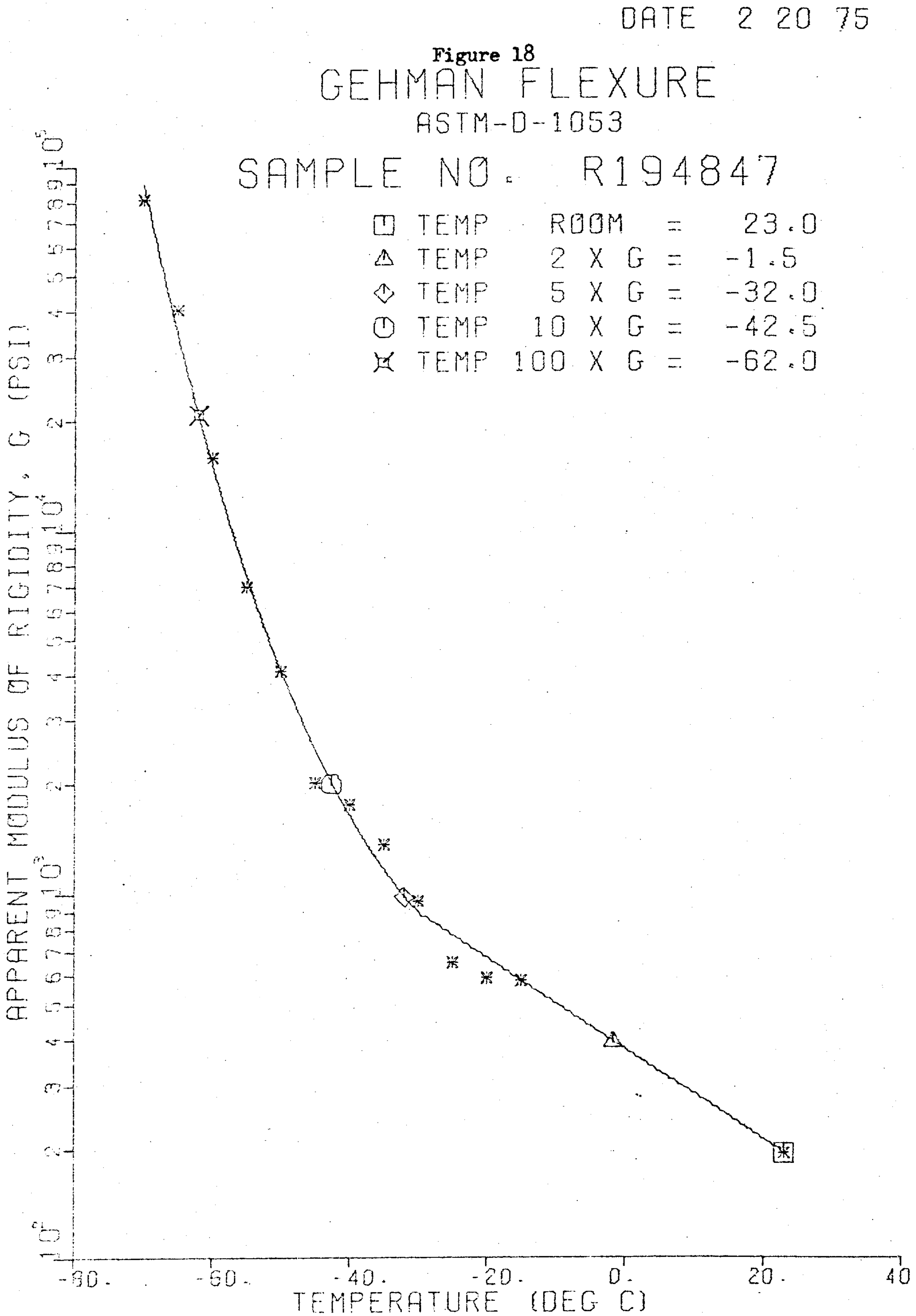

DATE 2 20 75
GEHMAN FLEXURE
ASTM-D-1053
SAMPLE NO. R194847
TEMP ROOM = 23.0
TEMP 2 X G = -1.5
TEMP 5 X G = -32.0
TEMP 10 X G = -42.5
TEMP 100 X G = -62.0
APPARENT MODULUS OF RIGIDITY, G (PSI)
TEMPERATURE (DEG C)
-80.
-60.
-40.
-20.
0.
20.
40.

TABLE XXVII
GEHMAN FLEXURE
ASTM D-1053
BLACK CODE WIRE

SAMPLE NO. R194847
DATE 2 20 75

A = 0.1250
B = 0.0820
MU = 3.1700
K = 0.1250

TEMP DEG C	X DEG,	G PSI
23.0	126.0	194.9
-15.0	79.0	581.5
-20.0	78.0	594.8
-25.0	74.0	651.5
-30.0	58.0	956.7
-35.0	45.0	1364.5
-40.0	37.0	1757.9
-45.0	33.0	2026.1
-50.0	18.0	4093.6
-55.0	11.0	6988.1
-60.0	5.0	15919.6
-65.0	2.0	40481.5
-70.0	1.0	81417.8

ROOM TEMPERATURE	23.0	GEHMAN FLEXURE	194.9 PSI
2 TIMES G	-1.5	GEHMAN FLEXURE	394.3 PSI
5 TIMES G	-32.0	GEHMAN FLEXURE	988.4 PSI
10 TIMES G	-42.5	GEHMAN FLEXURE	1995.5 PSI
100 TIMES G	-62.0	GEHMAN FLEXURE	20857.3 PSI

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The purpose of this investigation was to develop and evaluate phosphonitrilic fluoroelastomer compounds for O-ring hydraulic seal applications. Formulations were sought which would be serviceable in hydraulic fluids over the temperature range of -80°F to 400°F (-62°C to 204°C).

The polymer used in this investigation was a phosphonitrilic fluoroelastomer prepared in the Central Research Laboratories of The Firestone

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Tire & Rubber Co. and having the following formula: $(CF_3CH_2O)(HCF_2(CF_2)CH_2O)P = N_n$. The polymer contained sufficient cure sites to attain good curability with conventional peroxide curatives.

In addition to the low temperature flexibility indicated above the following target values were adopted for this investigation: Tensile strength - 1500 psi, elongation at break - 125%, 100% modulus - 800 psi, Shore hardness - 70, compression set (70 hrs.@ 300°F) - 20%.

This investigation was conducted under Contract No. DAAG46-74-C-0066 from the U. S. Army Materials and Mechanics Research Center, Watertown, Massachusetts 02172. The effects of reinforcing agents, vulcanization agents and stabilizers on compound properties were investigated. Efforts were made to optimize curing times and temperatures; stress-strain properties; hardness; compression set; tear and abrasion resistance; fluid, water, steam and acid resistance, and low temperature flexibility.

In addition, the Seal Group of Parker Hannifin fabricated O-ring seals from selected stocks and conducted dynamic extrusion and chew tests on the seals. These tests showed the best phosphonitrilic fluoroelastomer O-ring compound to be the following: Polymer K-17638 - 100.0 parts, Quso WR82 - 30.0 parts, Stan Mag ELC - 6.0 parts, Stabilizer - (8HQ)Zn - 2.0 parts, Union Carbide Silane A-151 - 2.0 parts and Vulcup R - 0.4 parts. This formulation afforded the best balance of stress-strain properties, hardness, compression set resistance, hydraulic fluid resistance and heat resistance. O-rings fabricated from this formulation should be serviceable for extended times over the temperature range of -70°F to 350°F (-57°C to 177°C).

In related studies experiments were conducted on the coating of stainless steel cable with phosphonitrilic fluoroelastomer compounds. A good quality coating of approximately 0.031" thickness was obtained by passing the cable through a crosshead extruder followed by vulcanization of the coating for 1 minute at 392°F (steam).

Phosphonitrilic fluoroelastomer compounds show limiting oxygen index (LOI) values of 50-60 depending on the type and level of filler incorporated in the compounds. These high LOI values add still another dimension to the applicability of phosphonitrilic fluoroelastomers in highly sophisticated environments.

This investigation has clearly established that phosphonitrilic fluoroelastomers have potential for applications demanding extreme low temperature flexibility, outstanding fluid resistance, good heat resistance and good dynamic properties. O-ring seals are one such application for which no existing commercial elastomer currently has met the full range of properties required.